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Evaporation Unit for a precursor for PECVD apparatus

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B2612 Elektrotechnika a informatika
B2612 Electronics and Information Technology

Jednotka pro odpařování precursoru pro PECVD aparaturu
Evaporation Unit for a precursor for PECVD apparatus

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Supervisor

Rozsah práce:

Počet stran	40
Počet obrázků	11
Počet grafů	10
Počet příloh	2

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30.4.2007 Jana Macků

Anotace

Cílem bakalářské práce je návrh a provedení elektricky vyhřívané jednotky pro řízené odpařování precursoru pro PECVD aparaturu. Práce obsahuje popis návrhu vypařovací jednotky a jejího praktického provedení. Experimentální měření charakteristik bylo provedeno v PECVD aparatuře. Takto byly určeny charakteristiky odpařovací jednotky pro umožnění jejího použití v laboratořích. Dále je poskytnuto srovnání s komerčně nabízenou termostaticky vyhhřívanou jednotkou.

Abstract

The aim of the BS thesis is the design and construction of an electrically heated unit for controlled evaporation of precursor for PECVD apparatus. The work contains a description of the design of the evaporation unit and its implementation. The experimental measurements were performed with a PECVD apparatus. Thus, the designation of the characteristics of the evaporation unit was done to enable the utilisation of the project in laboratories. Furthermore, there is a comparison with the characteristics of a commercially available thermostatically heated evaporation unit enclosed.

Acknowledgements

I would like to thank to my supervisor Prof. Petr Špatenka for his professional help, advice, suggestions and leadership. I want to acknowledge the collectives of both University of South Bohemia České Budějovice, Pedagogical Faculty, Department of Physics, and The Technical University in Liberec, Faculty of Mechanical Engineering, Department of Material Science for their willingness to help. I would especially like to acknowledge Ing. Jiří Cerman for his professional leadership during my training at The Technical University in Liberec.

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1. Introduction

The aim of my bachelor work is the design and construction of an electrically heated unit for controlled evaporation of precursor for PECVD apparatus, including the measurement of the characteristics of the evaporation unit to make it available for use in laboratories.

My work consists of a theoretical part outlining CVD and PECVD processes followed by the subsequent text remarking about some facts from the kinetic theory of gases, evaporation problematics and throughput, all vital to the resolution of the measurement.

The work contains a description of the design of the evaporation unit and its implementation. The main part of my work is the designation of the characteristics of the evaporation unit, which enable the utilisation of the project by other laboratories.

Furthermore, there is a comparison with the characteristics of a commercially available thermostatically heated evaporation unit enclosed.

2. CVD

Chemical Vapour Deposition (CVD) has become one of the most important methods of film formation and now constitutes a corner stone for modern technologies such as solid-state electronics. The reasons for the rapidly growing importance of CVD lie primarily in its versatility for depositing a very large variety of elements and compounds in the form of both vitreous and crystalline layers having a high degree of perfection and purity.

2.1 Fundamental aspects of CVD

Fundamental aspects of CVD cover reaction chemistry, thermodynamics, kinetics, transport phenomena and film growth, so the nature of the method is interdisciplinary. Thus boundaries of several classical fields should be crossed to understand the CVD processes.

CVD is based on the chemical synthesis of layers from vapour phase at a temperature near 1000^{o}C. Deposition apparatus for thin film production is comprised of the following basic components. Vapour matter generator containing metal (precursor), gas flow regulators, deposition chamber, heating, pumping and control and regulation system. It is possible to let almost any gas combination in the deposition apparatus.

CVD has plentiful applications, which could be categorised into insulators, elemental and compound semiconductors, conductors and miscellaneous types of materials. Although CVD is well elaborated, its utilisation is limited due to high temperature deposition process. CVD can not be applied where deposition temperature has to remain low to avoid heat degradation of a substrate.

2.2 From CVD to Other Thin Film Production Methods

The conventional (thermal activated) CVD uses thermal energy to activate the chemical reactions. However, the CVD reactions can be initiated using different energy sources. This has given rise to other variants of CVD methods such as plasma enhanced

CVD (PECVD) and photo-assisted CVD (PACVD), which use plasma or light to activate the chemical reactions. Other variants of CVD include metal-organic CVD (MOCVD), which uses metal-organic as the precursor rather than inorganic used conventionally, aerosol assisted CVD, which uses special precursor generation and delivery, flame assisted vapour deposition (FAVD), where flame source initiates the chemical reaction and heating of the substrate. Furthermore, there are other methods like electrochemical vapour deposition (EVD), chemical vapour infiltration (CVI), electrolytic coating, Physical Vapour Deposition (PVD), and many others.

3. PECVD

High energetic demand of CVD resulting from high deposition temperature, long operating cycle and temperature stabilisation as well as cooling down and ecology stimulated the development of plasma chemical methods to deposit thin layers. The physical chemical method enables the temperature to decrease by chemical reactions proceeded in an ionized atmosphere i.e. in plasma. This is why methods using plasma to deposit layers are called PECVD (Plasma Enhanced Chemical Vapour Deposition).

PECVD is a method for good quality deposited layers at temperatures near 500^{o}C. These are based on plasma processes in decreased pressure between around 10^{0} and 10^{3} Pa. One of the most significant advantages of this method is the diffusion of deposition, which enables the coating of complicated shaped substrates. Films became technologically important in such diverse areas as optical coatings, packaging, and in films in microelectronics.

3.1 PECVD Principle

PECVD uses electron energy (plasma) as the activation method to enable deposition to occur at a low temperature and at a reasonable rate. Supplying electrical power at a sufficiently high voltage to a gas at reduced pressures results in the breakdown of the gas and generates glow discharge plasma consisting of electrons, ions and electronically excited species. The vapour reactants are ionized and dissociated by electron impact, and hence generate chemically active ions and radicals that undergo the heterogeneous chemical reactions at or near the heated substrate surface and deposit a thin film. The temperature of the electron can be on the order of 20,000 K or higher, while the temperature of the vapour reactants may remain near room temperature, depending on the pressure at which the discharge is operated.

Activation of the operating compound when plasma is in direct contact with the surface of a precursor transmits part of the energy of the discharge to the substrate, which heats it up indirectly. The maximum approved temperature of the substrate at the end of the deposition limits the output power supply of the discharge.

3.2 PECVD Process

Plasma deposition is a very complicated process which involves a lot of phenomena from the area of physics and chemical plasma like elementary ionising and exciting processes, plasma interaction with solids, chemical processes in plasma and on the surface of a substrate.

Plasma deposition has not been understood comprehensively. Micro layer processing is yet based on the variation of macroscopic process parameters such as discharge supply, operating gas flow, pressure etc. Process is optimised according to the desired layer characteristics.

Available fundamental information about PECVD processes is limited and hence it is difficult to establish the relationship between the processing parameters and properties of the films because of the complexity of the PECVD reactions. Therefore, PECVD is limited to low-temperature applications where it cannot be met by thermally activated CVD.

The chemical reactions that occur during the glow discharges are complex and can be categorised into homogeneous gas phase collisions and heterogeneous surface interactions. Most commonly, the plasma chemical reactions occur in a compound of two different gasses, which react and create a layer of required chemical composition on the surface of a substrate. A lot of different organic, inorganic and metal-organic gasses can be used.

Nowadays, PECVD seems to be a perspective method that enables the growth of carbon nanopipes and nanofibres for medicine.

3.3 Types of Reactors

PECVD can be operated using either direct or remote modes. The direct PECVD reactors involve gaseous precursors, inert carrier gas and substrates placed in the plasma source region. During this deposition, substrates are placed directly on a heated

electrode. Surface of the substrates is fully exposed to ion bombardment thanks to emerged priming-grid voltage. Hence, direct PECVD is also convenient to clean a substrate surface immediately before deposition. This affects the adhesion of deposited layers positively, especially at low temperatures.

However, the remote PECVD methods generate plasma away from the deposition zone. This can avoid film damage caused by energetic ions and electrons in the plasma.

Direct current, high and low frequency or a microwave field can be used for plasma generation, all in quite a wide range of power level outputs. Usage of DC electrical field excludes deposition on dielectric substrates and formation of a dielectric layer, which can be made with HF supply.

3.4 Characteristics of PECVD Products

The films that are deposited by plasma reactions are usually amorphous in nature, with very little short-range structural ordering. The stoichiometry of the films can be made to vary in a controlled fashion by variation of dominant plasma parameters, such as reactant gas flow ratios. Because there is a range in film stoichiometry, it is to be expected, and is indeed observed, that electrical, mechanical and chemical properties can also vary.

3.5 Monitoring

The control of plasma-enhanced CVD processes is assuming increasing importance. There may be three main sections of control divided. Firstly, it is the field of process control probes, which help to monitor the plasma deposition process. This work, however, is not devoted to describe issues such as mass spectroscopy and optical spectroscopy, which detects gas phase intermediate species and monitors their concentration, and others. Secondly, there is the field of the control of macroscopic quantities, which are quite easy to regulate. Finally, PECVD control should be achievable by the use of film monitors.

There are various methods of film determination such as the measurement of film composition, adhesion, cohesion and hardness properties. For instance, the adhesion and the substrate-layer interconnection is measured with an indentation hardness test, and is a wide spread method for both organic and inorganic layers on metal, alloy, semiconductor, glass, mineral, and organic substrates. Calotest is a method used for layer thickness determination; its principle is the measurement of the annular area, which is cut into the surface. The Pin-on-Disc method determines the tribological properties like the adhesion, the abrasion and the friction factor.

3.6 System Requirements

Every glow discharge system for thin film production is comprised of the following basic components. They are the reactor or deposition chamber; power generation source, a possible impedance matching network to transfer the power more efficiently from the generator to the gas load, gas flow regulators, gas control section, pressure measurement plus other panel instrumentation.

Most plasma systems do not require critical high vacuum or pumping components. In general, the choice of the pumping system must be considered carefully based on the considerations of the pumping rate for all gases introduced and generated in the process, inlet flow rates, background contamination from residual gases, out-gassing and back-streaming.

Electrode design approaches for achieving the power to the plasma at the voltage level suitable for gas breakdown. Many variations are possible. The substrates, however, usually lie flat for heating purposes so larger ones are preferable. In different reactor geometry external electrodes can be provided.

3.7 Deposition of Titan Oxides

An Example application of PECVD could be Ti deposition. Nowadays, there is a lot of effort made on behalf of TiO_x thin layer development, as the optical, electrical

and tribological properties are outstanding. The characteristics of the layers depend on stoichiometry, deposition method and the conditions of the method. This indicates that the problematic is only going to be mentioned briefly. When developing new methods, it is momentous to consider the environment and health and safety. Metal-organic compounds minimise the risks, but more expensive in comparison with commonly spread Titanium Tetrachloride. However, outlining of the choice of the working compound is not intended.

The result of work with TiO_x is showing very good results. Friction of the PECVD layers proved to be better than those implemented with PVD. Thus it can find utilisation in the production of bearings for instance. This could be one of the reasons why the evaporation unit was tested for Titan Isoproxide, a prospective working compound.

4. Theoretical Background

It is not possible to fully understand the physical processes, which occur in applications of plasma without knowledge of the behaviour of gases on a microscopic scale, i.e. at the level of individual atoms, molecules, and charged particles. A branch of physics called the kinetic theory of gases describes the behaviour of gases on the microscopic scale, and the subject of statistical mechanics treats the implications of this behaviour in the macroscopic world.

4.1 Kinetic Energy and Temperature

When a set of atoms is put into an enclosure, the atoms will absorb energy from the environment, for example by radiation from the walls. The energy of gas is stored in the form of translational kinetic energy (and in the case of molecules, also in vibrational and rotational states). Because the gas molecules are in motion, frequent collisions occur both amongst the molecules and between the molecules and the wall, and these collisions cause a continual interchange of energy. It can be expected that the overall kinetic energy of the gas, and the average kinetic energy of each constituent molecule, depend on the absolute temperature T.

4.2 Evaporation, Condensation and Evaporation Rate

Elements of a matter liberate from its surface and make vapour of the matter in a volume. This effect is called evaporation for liquid phase and sublimation for solid phase Heat energizes the elements to release from the chemical linkage with other elements on the surface. The inverse process where molecules of a vapour after an impact link with the surface is called condensation and it is accompanied by heat release.

Evaporation rate is determined by the weight of a matter evaporated per one second from a surface of a set area at a known temperature. Evaporation rate can be designated from the total amount of particles evaporated per one second, which is equal to the amount of particles condensing per the same time on the same surface.

Total amount of the liberated particles does not depend on the fact, whether the evaporated molecules are drawn off or whether they condense on the surface again. If the volume above the surface that molecules evaporate from is confined, the concentration of molecules and pressure increase in this volume until equilibrium.

The number of condensing molecules can be designated from the number of effective collisions of molecules with the surface. Total amount of particles multiplied by the molecular mass equals the mass of evaporated matter. In case vapour is not drawn off from the system completely and its pressure increases to a certain value, some of this vapour condenses. Evaporation rate depends on temperature and on molecular mass of the particles.

The properties of a deposited layer are strongly dependent on the relative precursor concentration in the plasma discharge. . There is a proven dependence of electron concentration on metal-organic concentration in the deposition process. The deposition process is affected by many quantities such as power supply, substrate temperature, operating gas flow and relative precursor concentration in the discharge, which is the most difficult one to control. Measurement of partial pressure of the precursor is impossible because the precursor vapour tends to condense. Another problem is the low mobility of the precursor molecules.

The amount of particles, which liberate from the surface of the precursor in time, is crucial to determine the precursor concentration in the plasma discharge during the deposition process. The number of released molecules depends directly on the saturated vapour pressure and indirectly on the square root of the product of the evaporation matter molecule mass and temperature. Partial pressure is expressed in the equation bellow:

$$p = A e^{-\frac{B}{T}}$$

Where A and B are constants, which are available for some chemical agents only, p is partial pressure and T is temperature. The temperature dependence of concentration of the released particles in time is:

$$N = C S \frac{p}{\sqrt{MT}}$$

Where N is the released particles concentration, C is a constant, S is the evaporation area, p is the partial pressure, M is the molecule mass of the evaporation matter and T is the temperature of evaporation.

Partial pressure is difficult to measure, but its concentration is indirectly dependent on the evaporation temperature. Relative precursor concentration depends on evaporation or sublimation area, which is an unmeasurable quantity.

4.3 Gas Flow and Throughput

The reality in plasma processing is not a sealed enclosure but a steady state population of molecules being continuously fed by gas flow and continuously pumped by a vacuum system. In several plasma processes, in particular plasma etching and plasma deposition, the inlet gas is consumed and the need for the gas flow to replenish the consumption is apparent. On the other hand, in conventional sputtering there is no consumption of the etching gas. However, it is still needed to pump the process chamber to remove contaminants that might, as an example, be desorbed from the chamber walls and fixtures by the various types of fast particle bombardment present in plasma processing. A gas inlet flow is then needed to maintain the chosen operating pressure. At high pressures, the flow is viscous, either laminar or turbulent. At very high pressures, the flow is called molecular.

There are two main ways of describing the amount of gas flowing, for example, through a pumping port in a vacuum system. The pumping speed measures the volume of gas passing per second, but the number of molecules contained therein naturally depends on the pressure of that volume of gas. A frequently more useful term is thus the throughput Q, hence enhanced proportional to the flux of molecules passing. Throughput is alternatively known as flow rate, particularly when referring to the inlet rate of a reaction gas.

In a system of constant volume V, there is a gas of pressure p. Isochoric variation of pressure is then variation of throughput:

$$\mathrm{d}Q = V\,\mathrm{d}p$$

Pressure variation in time at constant volume of V and constant gas flow of S is

$$p = p_0 \exp\left(\frac{S}{V}\ t\right)$$

Where p is pressure and p_0 is initial pressure.

Leakage of a system causes linear increase of pressure in the system in time because flow rate from vicinity into the system is almost constant considering low pressure p in it.

$$Q = G\,(p_a - p) \approx G\,p_a$$

Where Q is gas flow per minute, G is vacuum conduction, p_a is atmospheric pressure and p is the pressure in the vacuum system. If pressure varies in a time of dt by dp isochorically, then this is the throughput.

$$Q = V\frac{dp}{dt}$$

Appropriate units for throughput and flow rate are of torr litre/second, pascal litre/second and the standard cc per minute (sccm), standard referring to standard temperature (0^oC) and standard pressure (1 atmosphere). It follows that:

$$1 \text{ sccm} = 2.69\ 10^{19} \text{ molecules per minute}$$

$$1 \text{ torr litre /second} = 79.05 \text{ sccm} = 2.13\ 10^{21} \text{ molecules per minute}$$

4.4 Measurement of Gas Flow Rate

The prerequisite is the knowledge of the volume of the chamber and of the pressure within it. The volume of the chamber can be reasonably estimated by physical measurement, but a much more accurate technique is to expand a small known volume of gas at atmospheric pressure into the evacuated chamber and note the resulting pressure.

To measure a given flow rate, the chamber is first evacuated and sealed. At time $t = 0$, the gas is allowed to flow into the chamber and the resulting pressure rise is noted as a function of time. This should result in a linear increase of pressure. As explained above, the pressure rise is indirectly dependent on square root of temperature.

5. Results and Discussion

5.1 Design and Implementation of the Unit

PECVD requires a source of the deposited matter. Especially for crystal like operating compounds, the best option is to ensure that the precursor is in liquid phase. Then the surface of the precursor, which the released molecules evaporate from, is fixed and the amount of evaporated particles can be designated. Subsequently, the vapour is let into the deposition chamber. The aim of this work is the characterisation of an operative evaporation unit for PECVD apparatus that can be utilised in laboratories. Its advantages should be easy usage, effectiveness and low production cost in contrast to evaporation units with commercially available heating, such as fluid circulatory heating. The heating of fluid circulatory heaters may also have adjustable parameters, but the usage is complicated and the purchase cost is very high.

5.1.1 Technical Drawing of the Unit

There is a scheme of the main part of the evaporation unit in Fig.1. There are more versions of the dimensions in testing. The volume of this one is sufficient for at least one deposition process and is meant not to waste the precursor. There is a thermocouple placed directly in the evaporation container and thus the measured temperature indicates the actual temperature of the precursor. The evaporation unit can be used in two ways; the vapour creation may proceed with the gas pipe either sealed or connected to a transport gas bottle. The transport medium is an inert gas, usually argon, to avoid chemical reactions with the precursor.

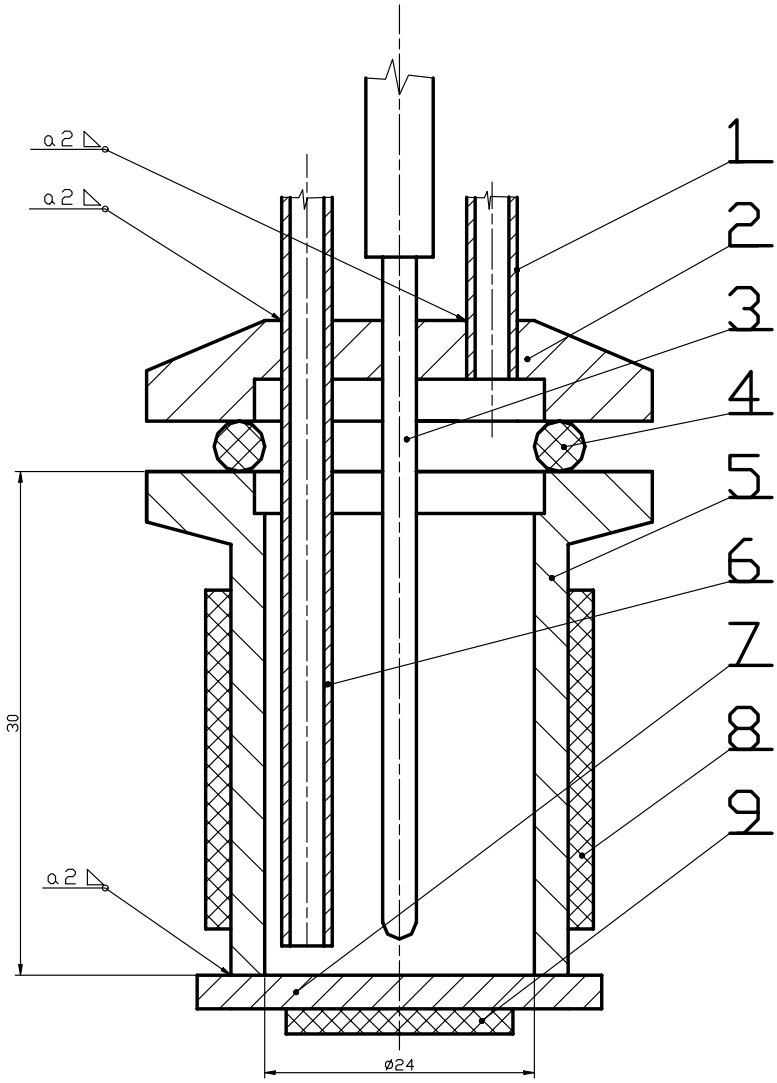


Fig.1 Scheme of the Evaporation Unit

1 Pipe for vapour conduction to the vacuum deposition chamber

2 Weld deposition modified container cover DN 25 ISO KF

3 Thermocouple

4 Neoprene seal DN 25 ISO KF

5 Container DN 25 ISO KF

6 Gas pipe

7 Bottom plate

8 Container cladding heater

9 Bottom plate heater

The evaporation unit was produced with the kind help of the laboratory at The Technical University in Liberec.

5.1.2 Heating and Temperature Regulation

Flexible heaters were chosen to heat the evaporating unit. The choice was based on the studies of the technical documentation [4] and [5] from a wide range of Silicone Rubber, Fibreglass and Kapton Insulated Flexible heaters. Silicone Rubber and Fibreglass Heaters (Fig.2) resist radiation, moisture, compression, weathering, vacuum, fungus, oils, solvents and chemical attack and are temperature rated up to 232^{o}C. They are lightweight, thin and flexible. They are available in 2.5 to 10 Watt densities, in either Etched Foil or Wire Wound Design, though etching was not efficient so wire binding was used. 115 V supplies the heater.

Fig.2 Flexible Heaters

However, he heater needed a temperature control. Ht 60 B-TA-RR-000 PID regulator (Fig.3), which is produced by HTH8 s.r.o., was chosen to control the heating. The same supplier offered monitoring software for the regulator. Thanks to this, program monitoring of the stabilisation of a temperature could be done to assure that the conditions in the evaporator were stable.

Fig.3 Regulator

These constants were used for the regulation output to control the temperature in the desired range. The regulation method is optional.

Proportional band Pb1 = 20.0

Integrative constant It1 = 5.0

Derivative constant dE1 = 0.1

Monitoring Program

Universal electrical connection of the regulator to a computer, where the monitoring program was installed, required a UC485 converter of RS232 to RS 485. In Fig.4, there is a scheme of the electrical connection.

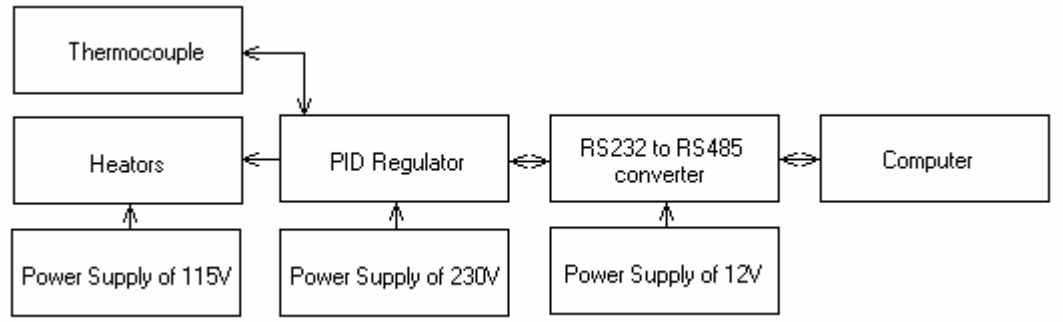


Fig.4 Scheme of the Electrical Connection

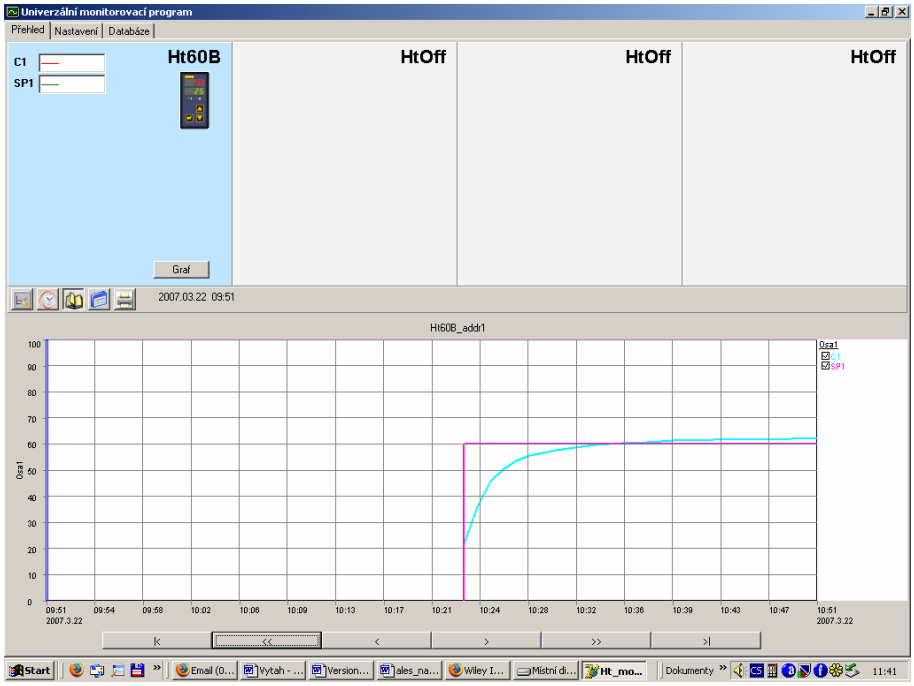


Fig.5 Window with a view of the program working

Fig.5 shows a window with a view of the monitoring program working. It is possible to monitor the current temperature of up to four regulators. When the electrical connection is done, it is necessary to detect the devices and then the monitoring is started. The program allows seeing the history, viewing the values in a table and exporting the data. There is also a possibility to adjust the view by changing the properties of the axis.

A rheostat strip was used to heat up the pipe conducting the vapour to the vacuum system. Since sedimentation in the pipe would depreciate the entire measurement and might even block the admission. The pipe was heated to a slightly higher temperature than the evaporation container. Power supply controlled the heating directly.

5.1.3 Components

Only components, which belong to vacuum laboratory everyday practise, were used in the experiment. Connection elements used for the experiment were small flange fittings suitable for this specific application thanks to their pressure range, tightness and temperature resistance of seals.

The needle valve was chosen according to the estimation of precursor inlet for the purpose of PECVD. The choice was based on experience with needle valves of different parameters. There is a picture of the needle valve in Fig.6. The producer of the valve offers a graph showing its flow coefficient at 37^{o}C, as shown in appendix 1.

Fig.6 Needle Valve

5.1.4 Isopropoxide Vapour

Titanium (IV) isopropoxide vapour, which is metal-organic, was used as the source of titan for PECVD. Its chemical formula is shown in Fig.7. The exact chemical term is tetrapropyl orthotitanate. This compound was chosen according to the application (outlined in 5.4). It is neither environmentally unsound nor corrosive.

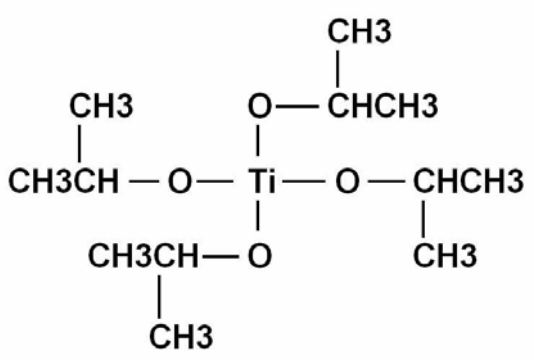


Fig.7 Chemical Formula of the Structure of Titanium (IV) Isopropoxide.

Precursor is usually evaporated at 60°C. The process of deposition is controlled by the regulation of the decomposition of precursor and the characteristics of a deposited layer by temperature and watt power changes.

5.1.5 PECVD apparatus

RF PECVD apparatus, which is from the laboratory at The Technical University in Liberec, was used. Its schematic diagram is shown in Fig.8. The apparatus is equipped with a shower inlet for vapour admission, which is connected to the pipe leading from the evaporator. The shower inlet is necessary to assure that the low mobility precursor molecules spread equally in the discharge during a deposition process.

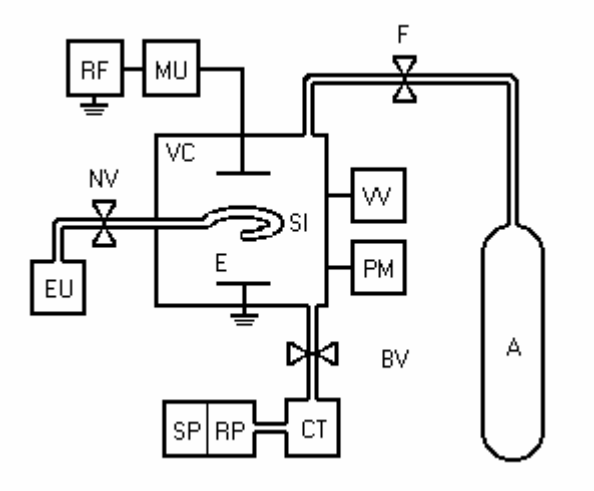


Fig.8 Scheme of the apparatus:

VC vacuum chamber, EU evaporation unit, NV needle valve, SI shower inlet, E electrodes, VV venting valve, PM Baratron pressure meter, BV butterfly valve, CT cryogenic trap, RP rotary pump, SP Root's pump, MU matching unit, RF radio frequency generator, F flow meter, A Argon

Fig.9 Apparatus where the experiment was performed

The apparatus during the experiment is shown in Fig.9. The central part of the system is the body of the deposition vacuum chamber. There is the evaporation unit, pressure probe, venting valve and a window for plasma discharge observation connected. Underneath the chamber, there is the suction branch of both rotary and root's pump with cryogenic trap. In the upper right corner, you can see the Matching Unit, which is used between the RF generator and the glow discharge. The purpose of this network is to increase the power dissipation in the discharge, and to protect the generator. And in the lower right corner, there is the regulated power supply unit for plasma (voltage transformer, ACG-3 RF supplier, Wattmeter 50 Ohms - plug-in elements determine power and frequency range).

5.1.6 Implementation

The evaporation container and its heating, the thermocouple, the pipe and its heating, the needle valve and the chamber flange connection are all included when using the term Evaporation Unit as can be seen in Fig.10

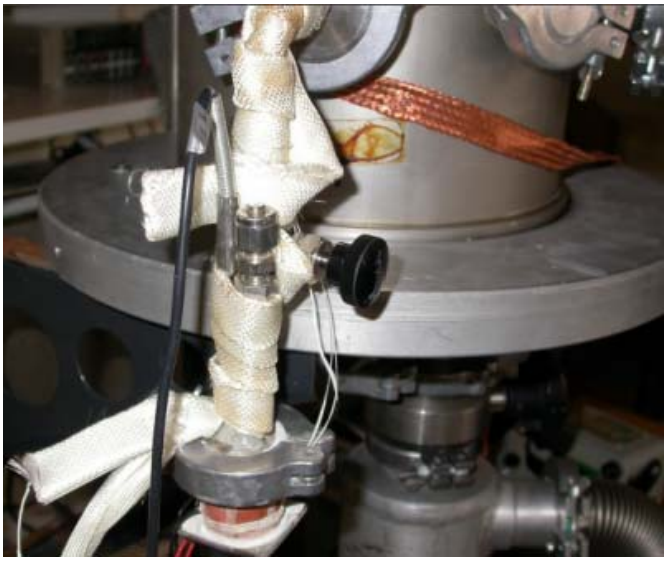


Fig.10 Detail of the evaporation unit arrangement

5.2 Experiment Description

Measurement of the Throughput of the Needle Valve

Measurements in a vacuum system can be done in specified conditions only so that the results are valid. Precisely designated throughput is the most important specification of the conditions. Therefore, basal throughput of the apparatus was designated before and after every measurement of the throughput of the valve at a specific temperature of the vapour. It can be presumed that the gross flow rate of the apparatus is linear in time, as the approximate duration of the measurement for each temperature was constant.

The apparatus was rigorously cleaned before every particular experiment, which could only start after a long period of pumping. Then the apparatus was provably prepared for the experiment from the view of pressure conditions. Meanwhile, the evaporation unit with the precursor inside was heated up to a desired temperature.

The experiment was performed with help of RF argon discharge. Since the precursor was evaporated at a temperature that was higher than room temperature, sedimentation prevention was needed. The discharge of 200W heated up the chamber to avoid sedimentation of precursor on the cold walls of the apparatus, which positively affected equal evaporation from the matter in the chamber and pressure deviation.

Because the pressure in the container of the evaporation unit was very high, the needle valve had to be fully opened to level out the pressure difference. After the balance was reached and the gross pressure in the apparatus decreased to the initial values of the experiment, the throughput of the fully opened needle valve could be measured. Thus the characteristics of the valve were measured from an opened to closed admission.

Throughput measurement can be done in two different ways as described in the theoretical part of this work. In this experiment, the time of pressure growth was measured. The dimensions of the apparatus volume were measured physically. Then the

pressure variation with time and the volume were calculated into values for throughput expressed in sccm, which are demonstrated in the graphs.

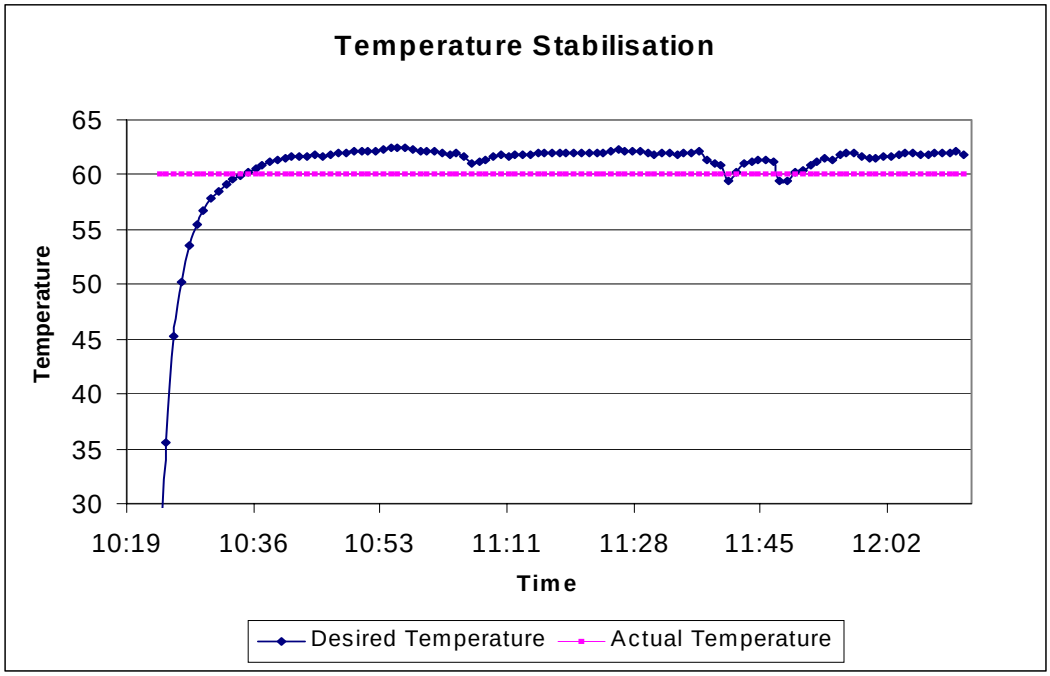
The experiment had to be repeated several times to guarantee the accuracy of the results. The following turns of the valve were measured in the same manner. The pressure in the apparatus was decreased and the needle valve was turned to a coveted position. When the pressure levelled out, the pressure variation with time could be measured again.

5.3 Measurement of the Precursor Flow

Firstly, I would like to present an example graph showing the regulation of the temperature in time, as the throughput is indirectly dependent on temperature. The graphs show the entire measurement line for 60^{o}C, since this is the temperature at which the precursor is conventionally evaporated. Subsequently, the graphs show the results for the other temperatures measured.

In this case, the temperature stabilised in ten minutes at a slightly higher temperature than desired. Heat transmission from the pipe leading into the deposition chamber caused this, since the heating of the pipe was only regulated by the power supply and the deposition chamber was heated up by an argon plasma discharge, both to prevent sedimentation.

Changes shown in Graph 1 are affected by the opening of the needle valve, which caused a rapid change of the conditions within the evaporation container, especially the evaporation. At 11:02, the needle valve was opened and the measurement started. At 11:37, an argon plasma discharge was lit to clean the chamber from sediments and support desorption, which caused higher evaporation from the entire surface including the surface of the precursor within the evaporation unit, as the needle valve was not closed for this process.



Graph 1 Regulation to 60°C

Fig.11 shows the stabilisation of the temperature displayed by the monitoring program. The period shown is limited and shows the first 20minutes of the process, which correspond to the initial part of Graph 1.

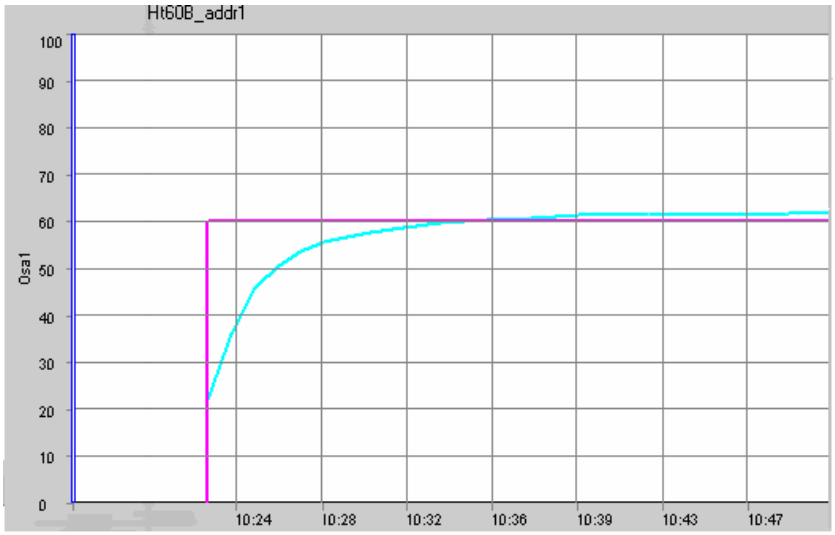
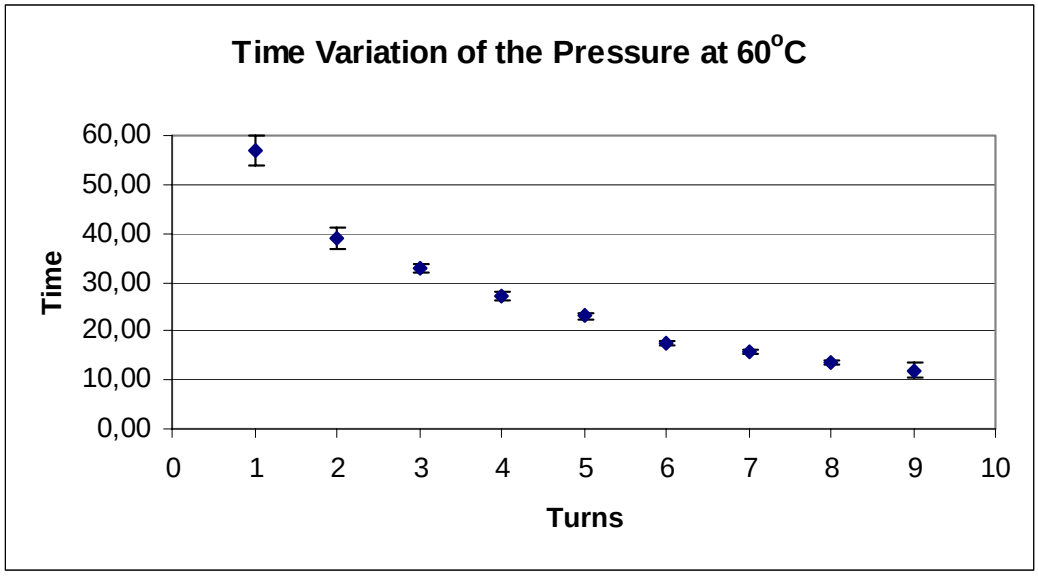


Fig 11 Temperature Stabilisation

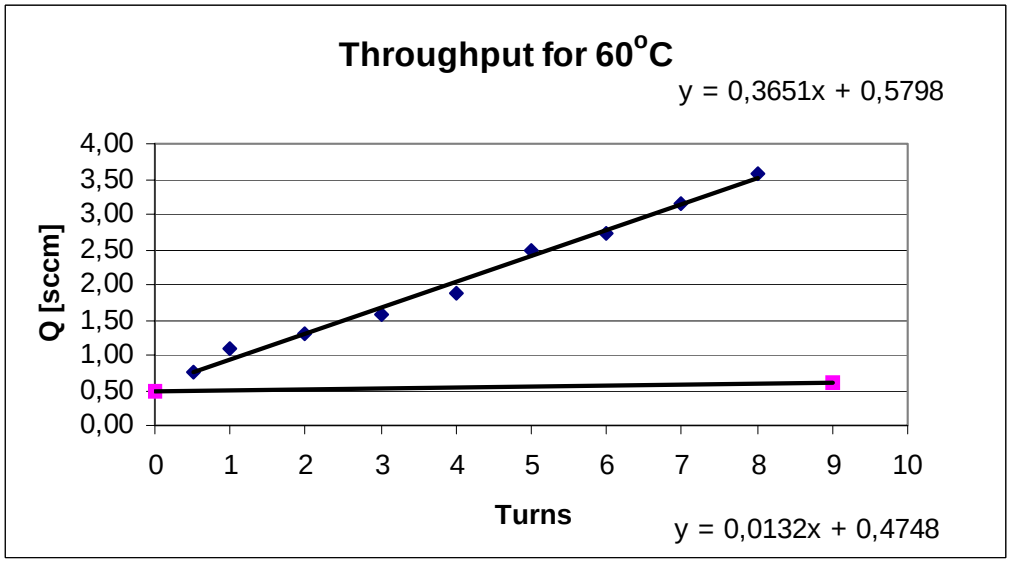
It can be seen that the temperature stabilisation was quite fast. Decisively fast enough in terms of vacuuming that has to forego the deposition, as the duration of this process is far longer.

Secondly, Graph 2 shows the measured time variation of the pressure for each turn of the needle valve at the temperature of 60°C. The following graphs are based on the data which can be seen in the appendix. As described above, the measurement started with the valve in the open position.



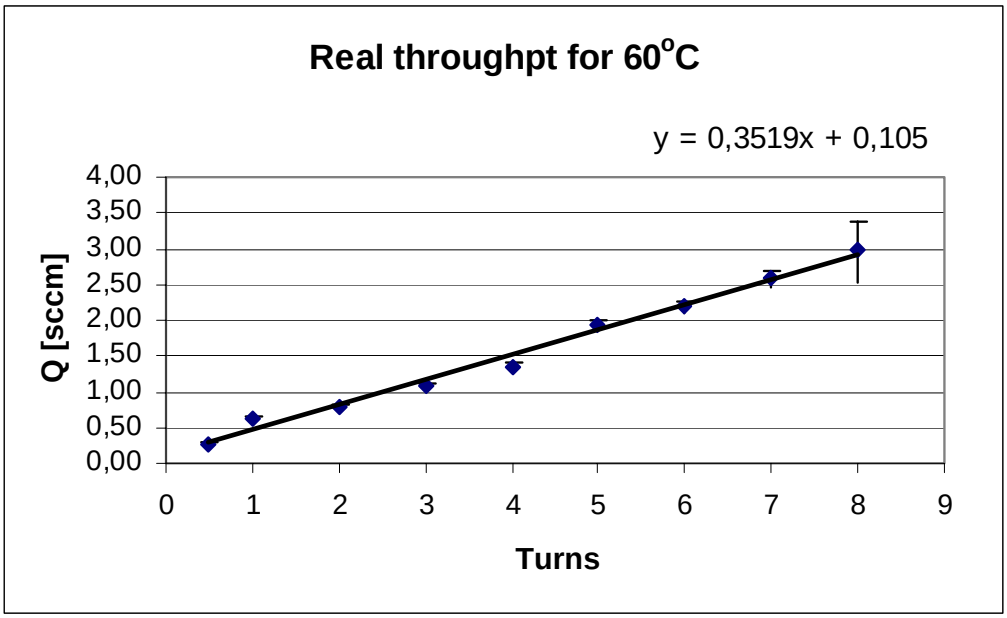
Graph 2 Time Variation of the Pressure at 60°C

As mentioned above, the gross throughput of the apparatus may be considered to be linear in time. Duration of the measurement for each turn of the needle valve may be constant. Thus, the gross throughput of the chamber may be subtracted linearly from the measured throughput of the needle valve.



Graph 3

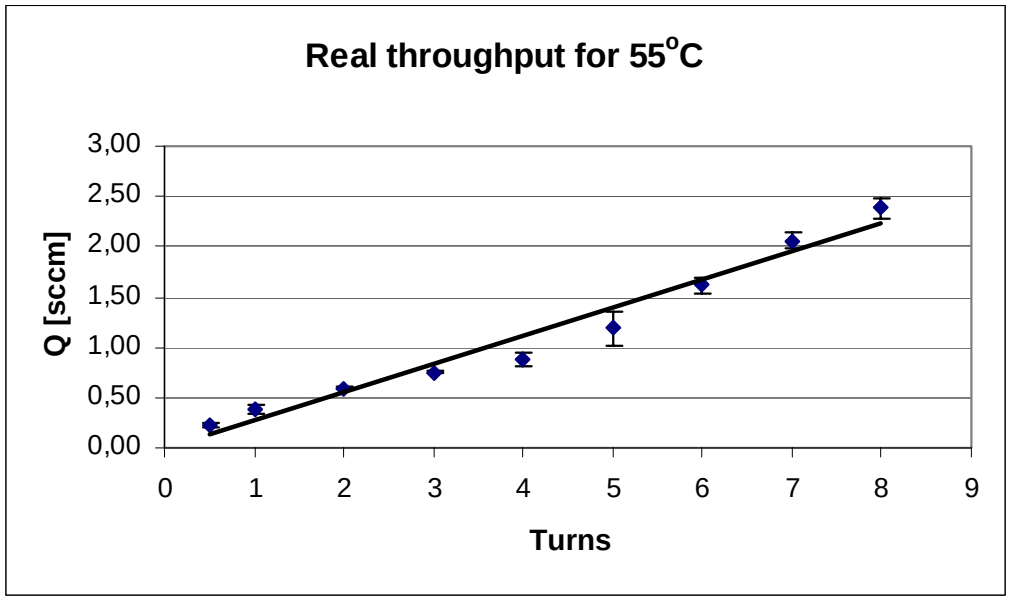
The throughput of the needle valve and the gross flow rate of the vacuum system at 60°C are shown in Graph 3. The temperature of the precursor reached 60°C in 10 minutes and remained between the temperatures of 59.6 and 62°C for the entire duration of the experiment.



Graph 4

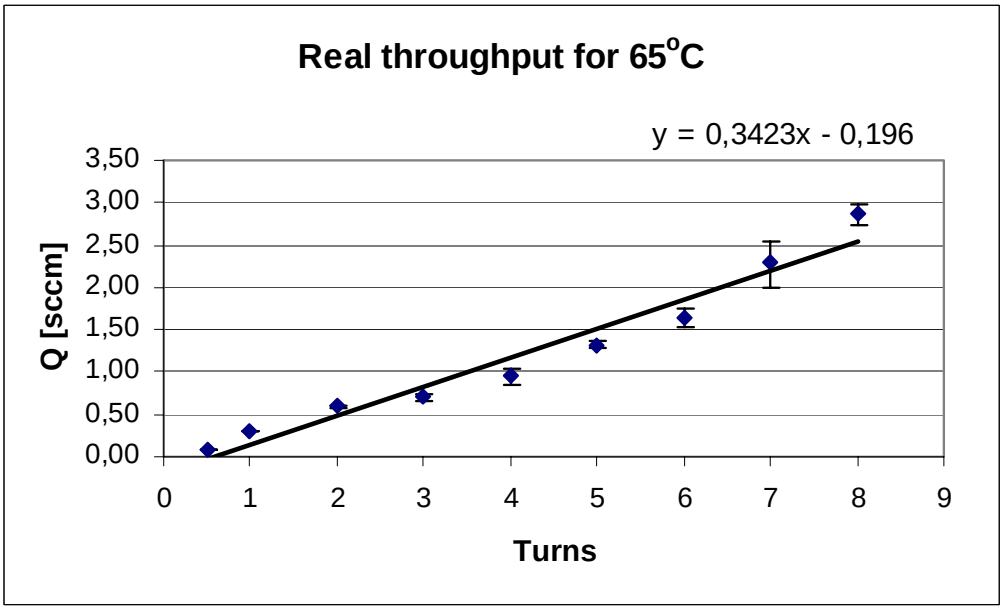
The real throughput of the valve at 60°C after the subtraction of the gross flow rate curve is shown in Graph 4. The curve fitted through the values is linear so the throughput depends on turns of the needle valve linearly, which fulfilled our expectations and also confirmed the needle valve flow rate properties. See the appendix for the characteristics provided by the supplier.

Graphs showing the throughput for temperatures of 55°C, 65°C and 80°C follow. The throughput of the needle valve and the gross flow rate of the vacuum system for these temperatures are shown in the graphs appended.



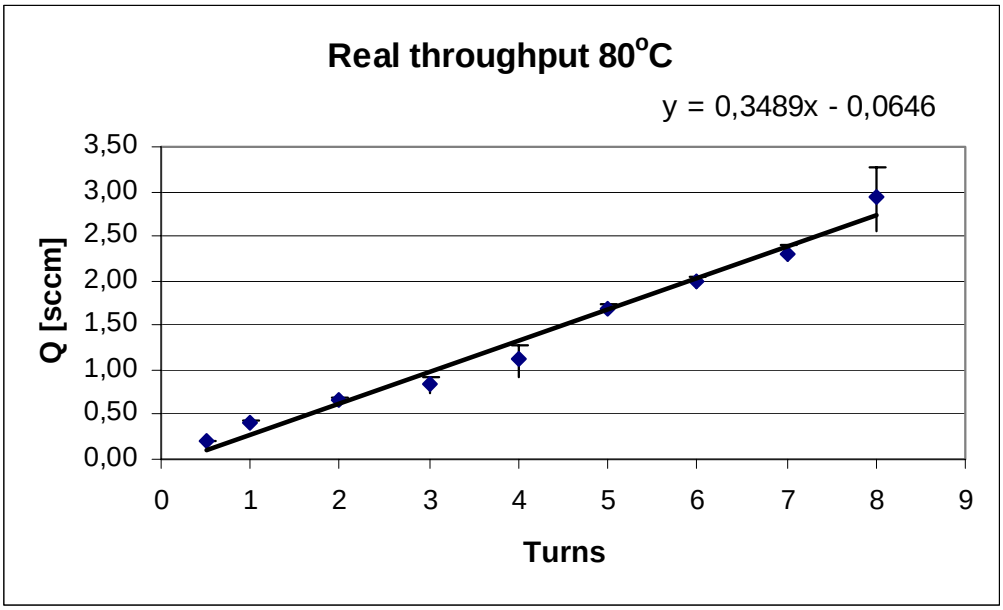
Graph 5

The real throughput of the valve at a 55°C after the subtraction of the gross flow rate curve is shown in Graph 5. The curve fitted through the values is linear.



Graph 6

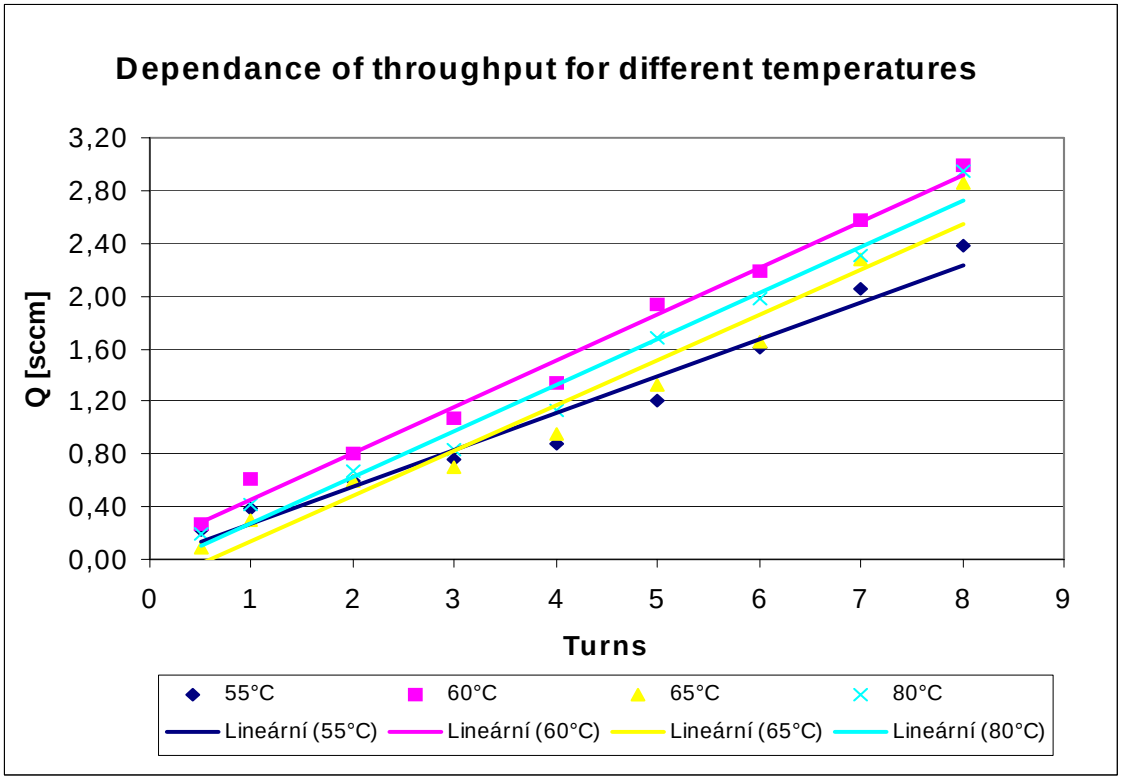
The real throughput of the valve at a 65°C after the subtraction of the gross flow rate curve is shown in Graph 6. The dependence is linear.



Graph 7

The real throughput of the valve at 80°C after the subtraction of the gross flow rate curve is shown in Graph 7. The curve is linear.

Finally, the subsequent Graph 8 shows the throughput for all temperatures.

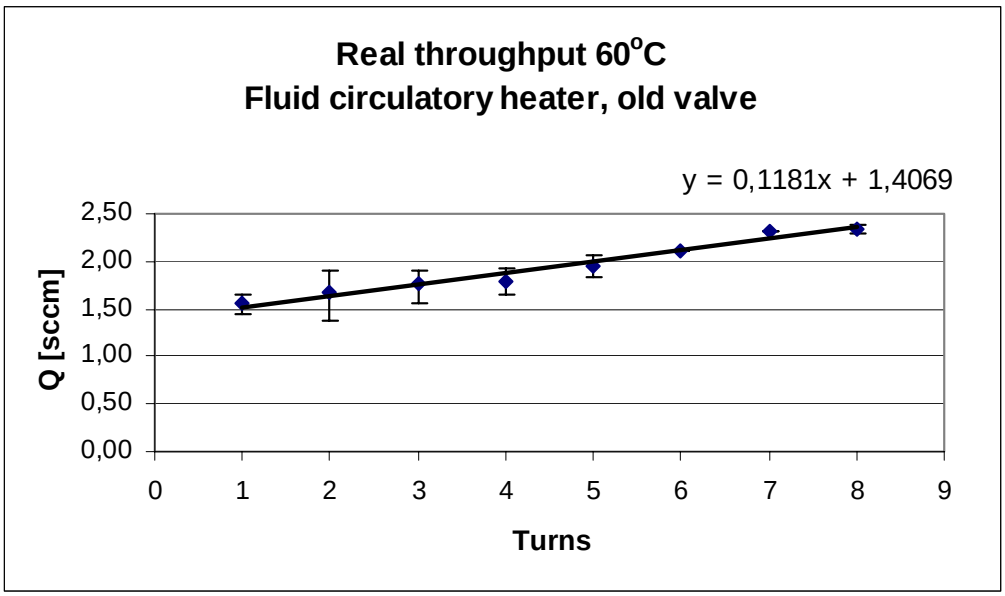


Graph 8 Lineární means linear, a Czech program used

The experimentally measured dependence is linear. The temperature relation is very slight and is not provable according to the graph above. That is because the measurement was done within quite a narrow temperature variance. The theoretical part outlined that the temperature dependence is slight because the relation for throughput depends indirectly on the square root of temperature.

Throughput of the needle valve is affected by the needle valve properties at most and thus it was sufficiently linear.

Furthermore, there is a graph showing the characteristics of the circulatory heated evaporation unit with an ethylene glycol carrier.

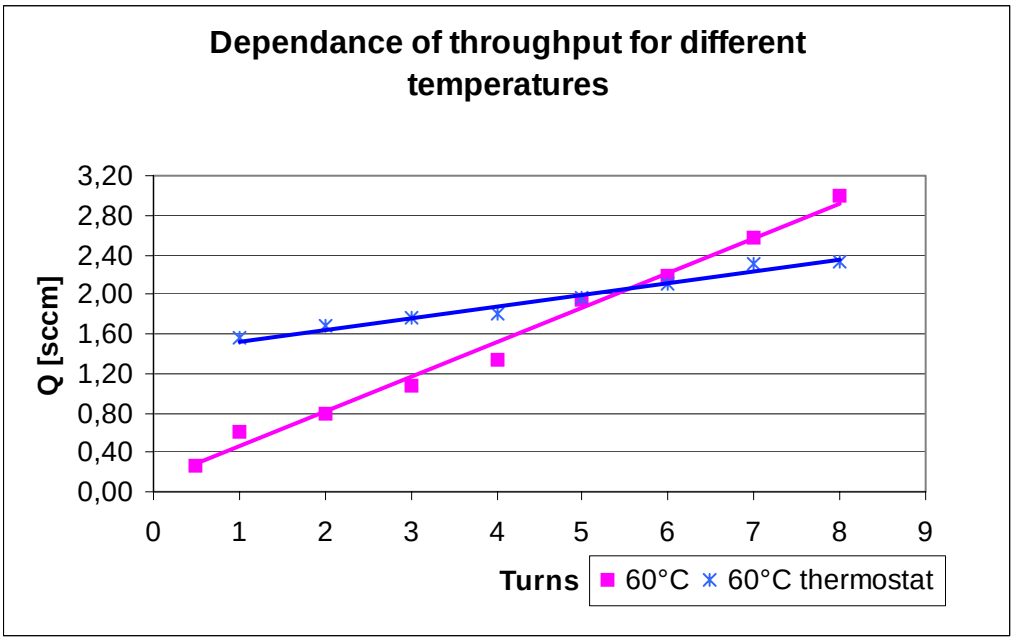


Graph 9

The real throughput of the valve at 60°C after the subtraction of the gross flow rate curve is shown in Graph 9. The curve fits linearly through the values of the throughput dependence on turns of the needle valve.

In Graph 10, there is the flow rate comparison of the evaporation unit heated electrically and the evaporation unit heated by a commercially offered fluid circulator,

which has an ethylene glycol carrier and is used in the laboratory at The Technical University in Liberec where my training took place.



Graph 10

The slopes of the curves in Graph 10 differ.

The reason might be that the volumes of the evaporation units differ. The volume of the electrically heated container was of 14.7 cm^3 and the volume of the fluid circulatory heated evaporation unit was 75.4 cm^3 which is roughly five times bigger. The evaporation area of the thermostatic evaporation unit is of about 2.5 times larger than the electrically heated one. The amount of the evaporated particles depends on the evaporation area, but the saturated vapour pressure for the precursor is constant.

If this held true, the curves would originate in the same place and the slopes would be different, which is not the case in this graph. Therefore, I exclude this reason.

The reason why the slopes of the curves are different and why they cross is probably because the needle valve, which was used for the measurement in the case of the fluid circulatory heater, was not the same. Its parameters differ due to the different calibration of the needle valve, although the ordering number was the same. This hypothesis can be confirmed by an additional measurement.

Comparison of the Temperature Stabilisation

The measurement of the temperature stabilisation was performed with containers of different volumes containing the same precursor.

In case of the fluid circulatory heated evaporation unit, the temperature of the carrier stabilised at 60^{o}C in 6 minutes, thermal variation was in tenths of a degree centigrade. The temperature of the fluid carrier was measured. The temperature stability of a fluid circulatory evaporator is very good but it is probable that it also does not assure absolute stability inside because rapid evaporation can cause temperature decrease of the liquid precursor.

The stabilisation of the precursor temperature takes about 10 minutes in the case of the electrically heated evaporation unit. At low temperatures (like 55^{o}C), the stabilisation takes slightly longer and is not as precise as at higher temperatures, independent on the selection of a regulation method. Generally, the temperature stabilisation was good. The recorded temperature deviations showed the situation within the container precisely because the thermocouple in the container was in contact with the precursor.

On the whole, the temperature stabilisation of both the electrically heated evaporation unit and the fluid circulatory heated unit is the same. The implemented electrically heated unit is highly efficient for the purpose of PECVD precursor preparation.

6. Conclusion

Firstly, the evaporation unit was assembled. The electrically heated evaporation unit operates well. The temperature stability is good and sufficient for the purpose. It can also be improved by appending insulation, which would decrease the energetic loss.

Secondly, the characterictics of the needle valve were measured and its properties determined. The dependence on temperature and its deviations is almost negligible for the purpose of PECVD. Thus the precursor can be regulated by the choice of the needle valve.

Furthermore, the comparison with the characteristics of a commercially available thermostatic heater proved that the electrical heating operates practically the same. The fact that the composition of the electrically heated evaporation unit embodies the thermocouple directly inside of the evaporation unit is its advantage, because the thermocouple measures the temperature of the liquid precursor, which helps to monitor the PECVD process.

Finally, the purchase cost of this evaporation unit is one tenth lower and the implication and the subsequent utilisation are much easier.

7. References and Bibliography

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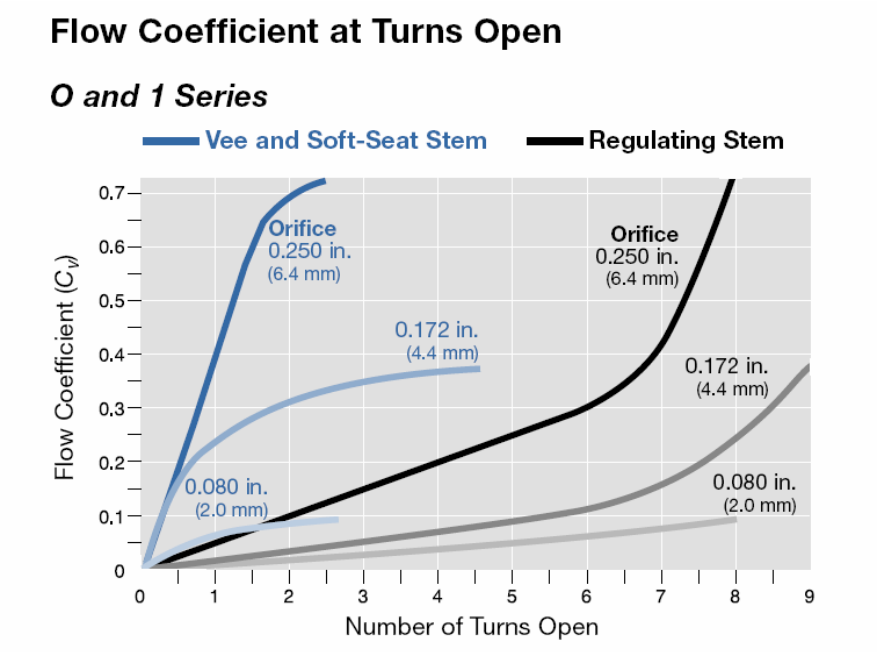
[7] www.swagelok.com - Catalogue 2007

8. Appendices

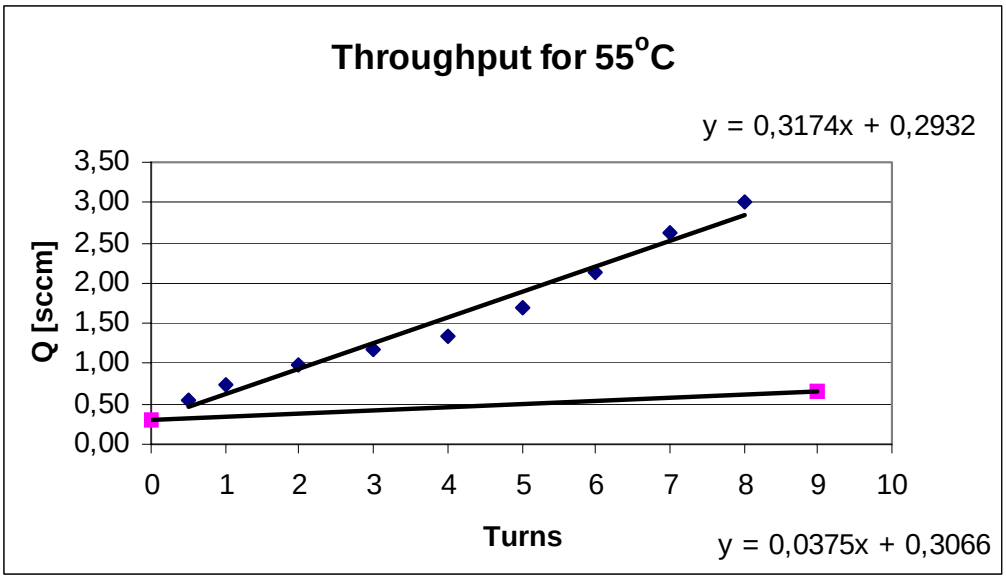
8.1 Characteristics of the Needle Valve

Flow Data offered by Swagelok, the light grey line shows the behaviour of the needle valve (SS-1RS6MM) used in the experiment.



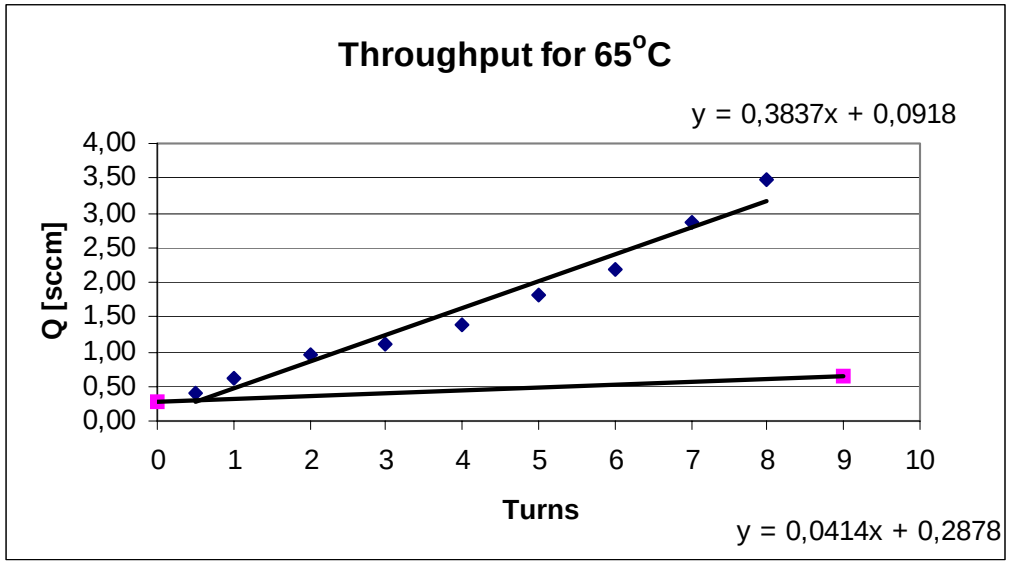
8.2 Graphs

Graphs for the throughput of the needle valve and the gross flow rate of the vacuum system for the temperatures of 55°C, 65°C and 80°C follow. The labels of these graphs correspond to the labels used for the graphs in 5.3. The errors of the gross throughput never exceeded 5% and the errors for the throughput of the needle valve are shown in the graphs after the subtraction of the gross flow rate.



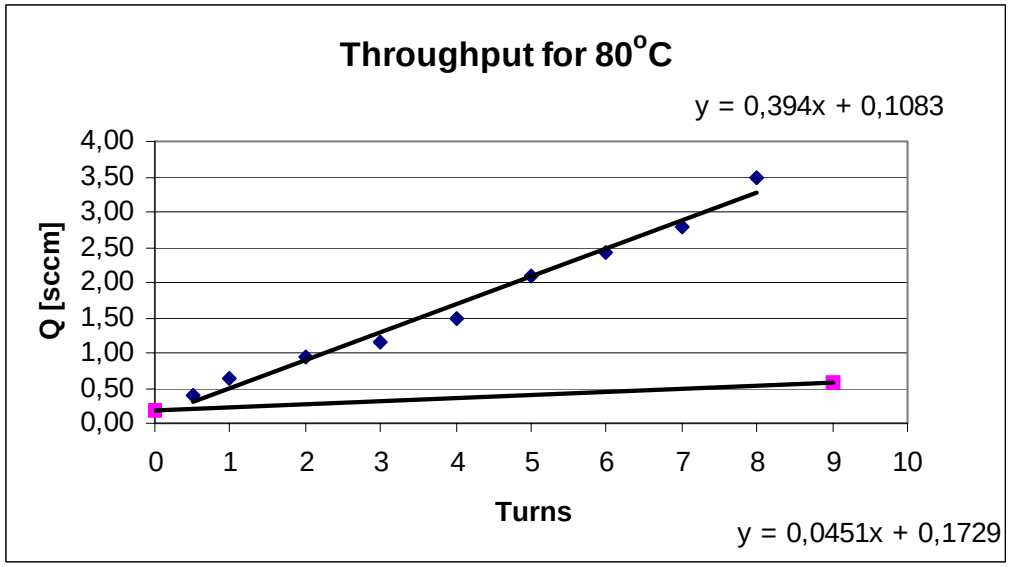
Graph 5b

Graph 5b shows the throughput of the needle valve and the gross flow rate of the vacuum system at 55°C. The temperature of the precursor reached 55°C from room temperature in 13 minutes and remained between the temperatures of 54.5 and 55.5°C for most of the measurement duration. The temperature lingered between 54.2 and 56.3°C during the entire process.



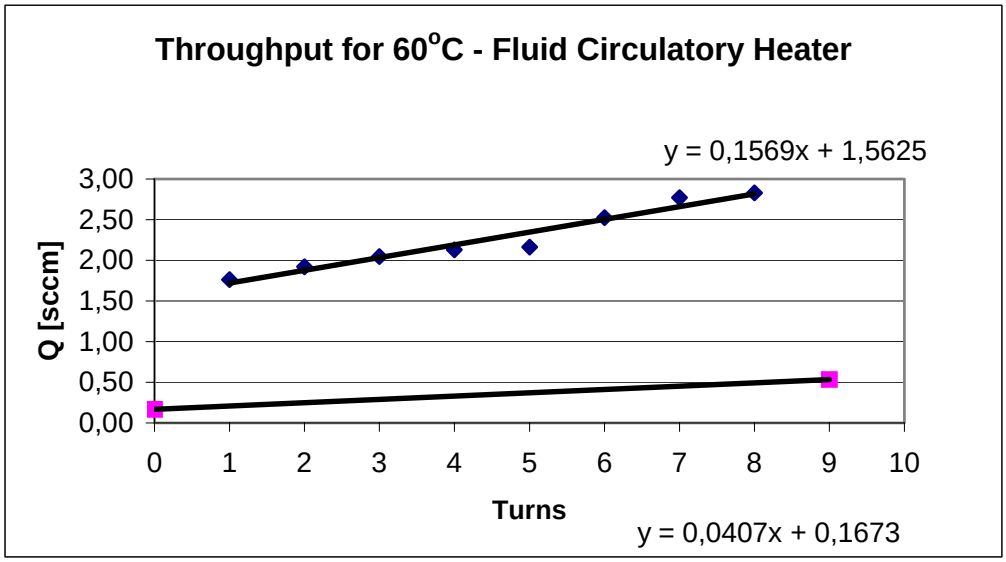
Graph 6b

Graph 6b shows the throughput of the needle valve and the gross flow rate of the vacuum system at 65°C. The temperature of the precursor reached 65°C in 10 minutes and remained between the temperatures of 65.2 and 65.5°C for most of the measurement duration. Extremes appeared due to argon discharge lighting, but the temperature lingered between 64.5 and 65.6°C during the entire measurement.



Graph 7b

Graph 7b shows the throughput of the needle valve and the gross flow rate of the vacuum system at 80°C. Temperature stabilisation during this measurement was very good. The temperature of the precursor reached 80°C in 9 minutes and remained between the temperatures of 79.4 and 81.0°C for the entire measurement duration.



Graph 9b

Graph 9b shows the throughput of the needle valve and the gross flow rate of the vacuum system at 60°C.