The Vacuum Science Group at Daresbury Laboratory is part of the UK’s Accelerator Science and Technology Centre, ASTeC. Members of the group have a wide experience in the design and operation of large vacuum systems for particle accelerators. The Group's main strengths lie in the development and use of advanced modelling techniques for vacuum system design, knowledge and understanding of specific particle accelerator vacuum problems as well as Ultra High and Extreme High vacuum technique. We use our skills both in the support and improvement of existing accelerator facilities and in the design and specification of new facilities.

The group is proud of its state of the art vacuum science laboratory and instrumentation laboratory. These laboratories are used to run an advanced programme of R&D as well as to provide the necessary vacuum facilities to underpin ASTeC programmes.

The principal areas of activity include:

- Investigating the properties of Non Evaporable Getters (NEG) and novel coatings for vacuum applications.
- Investigating the outgassing characteristics of materials for use in vacuum.
- Developing the techniques for producing thin films on vacuum vessel walls.
- Development of XHV technique for Photoinjectors and SCRF.
- Development of the materials used as photocathodes and processes involved in improving their performance.
- Vacuum metrology and calibration service.
- Investigating new cleaning techniques for UHV and XHV applications.
- Advanced modelling and design of vacuum systems.
- Evaluation and development of vacuum equipment including pump speed measurements.

In addition, members of the group are actively involved in training and education in the field of vacuum science and technology, paper reviewing for peer-review journals, organising vacuum conferences, workshops and other events, having links with a number of universities and departments as well as active involvement in the work of the International Union for Vacuum Science, Technique and Applications (IUVSTA), the American AVS and the vacuum group of the Institute of Physics (IoP).
The ultimate pressure in any vacuum system is basically limited by two factors:

1) Pumping Speed
2) Outgassing rate of the materials inside the chamber

Normally it is extremely difficult to increase the pumping speed by several orders of magnitude, therefore the most effective technique for any vacuum system is to try and reduce the outgassing rate of the materials.

This outgassing system enables the characterisation of outgassing rates using a measurement technique called the ‘throughput’ or ‘conductance’ method. The measurement of these outgassing rates allows the vacuum scientist to determine which materials are suitable for particular applications or pressure regimes. Materials with low outgassing rates would typically be used in construction of vacuum systems which can achieve <10⁻⁹ mbar, for example, particle accelerators. These materials are deemed to have low outgassing rates because they do not have large ‘sinks’ of gas and they can be conditioned for use in vacuum. Such materials are; stainless steel, titanium, aluminium, glass, ceramics and many other metallic elements.

Materials with high outgassing rates are generally non-metallic type materials, examples are, ceramics, porous metals, plastics, wood, liquids and paper. These materials whilst being large ‘sinks’ of gas are also difficult to condition for use in vacuum.

The Cleaning Outgassing Facility has been used to determine the outgassing rate of a whole range of materials used in many different applications, examples are:

1) Particle accelerators, electron, neutron and proton machines
2) Telecommunications
3) Satellites

This system has been used in recent years to assess the cleaning efficiency of both water and solvent based cleaners. To help achieve low outgassing rates some form of surface conditioning is required, this can be done by cleaning the surface of the material to be inserted into the vacuum system. The results of the study have been published and the overall conclusion was that solvent based cleaners are far more effective cleaning materials for particle accelerators than water based cleaners.
Surface science is an integral part of many aspects of science. It can be used to study a wide range of things, including chemical reactions, catalysis and surface structures. This allows scientists to understand the mechanisms behind what actually happens in Physics, Chemistry and Biology.

There are two very important programs of work in the vacuum science laboratory, one being the NEG coatings program, the other being the photocathode program. In each of these areas surface structure is fundamental to improving coating performance, quantum efficiency measurements, activation processes and many other aspects of this research.

The system shown has the following capabilities:
1) X-ray Photoelectron Spectroscopy (XPS)
2) Low Energy Electron Diffraction (LEED)
3) Ultra-violet Photoelectron Spectroscopy (UPS)
4) Temperature Programmed Desorption (TPD)

Each of the techniques listed can be used to provide different information that helps the scientist draw conclusions or formulate further experiments to confirm theories.

The surface science system forms an integral part of a collaboration between Liverpool University and the ASTeC Vacuum Science Group and demonstrates the Cockcroft model of universities and national laboratories working together. The system is essential to scientists and Ph.D. students and, is certain to be a critical arm of the groups research activities in the future.
This is a specially designed versatile coating system which can produce novel coatings with improved corrosion resistance, hardness, wear, catalytic performance, optical properties, thermal stability, biomedical applications and getter properties.

There are two main operation modes:

1. **Fixed carousel**

   Magnetrons A, B and C may be used separately, in pairs, or, together to produce a wide range of multi-element coatings. The ion beam can also be used prior to coating, to clean the substrate, or, during film deposition to modify film microstructure and density.

2. **Rotating carousel**

   All substrates can now be coated with material A, B, C and D from the magnetrons to produce complex multi-layer coatings, such as the MAX phases which can withstand temperatures above 1000°C.

   Film deposition with a planar magnetron system allows operation over a wide range of pressures, target compositions and deposition rates which allows better optimisation of the coating performance.

   The deposition chamber itself comprises four magnetrons and an ion source to clean the substrate surface prior to deposition. Only one magnetron is used for the deposition, but one needs to bear in mind that the configuration with two magnetrons in the chamber forms a partially closed magnetic field configuration. The base pressure is $5 \times 10^{-8}$ mbar. In order to provide an impurity-free environment the depositions are performed in high purity krypton, normally at an indicated chamber pressure of $2 \times 10^{-3}$ mbar.

   For NEG coating deposition the magnetron had a 70 mm diameter Ti-Zr-V alloy target with equal atomic concentrations of each element but other compositions can easily be used. The substrate and target were sputter-cleaned prior to deposition. Deposition can be carried out in DC or pulsed DC mode and the deposition power used was varied up to 200 W.
Pumping Speed Dome Test Facility

The Fischer-Mommsen test dome meets an ISO standard for methods of measuring the performance characteristics of a range of ion pumps and turbo-molecular pumps. Using this facility we are able to measure the total pressure and the volume rate of flow (pumping speed) for each individual pump.

The test dome is a circular cross-section with two chambers of a known volume, separating the upper and lower chamber is a thin flat plate with a small aperture (conductance) in the centre. Each chamber has a calibrated hot-cathode ionization gauge for measuring pressure, p1 and p2. An RGA (residual gas analyser) is also attached to the test dome to study the gas partial pressures in the system and having the facility for leak detection. A number of test gases can be introduced into the top chamber via a leak valve at a controlled rate, the manifold at the front of the facility enables a selection of gas species of high purity.

The pumping speed test facility in the vacuum science laboratory is used for a number of reasons, it enables us to gain knowledge and experience of new products on the market, make comparisons to the manufacturers data and as part of the process provides us with a valuable database for a whole range of pumps that may be utilised in future applications.

Calibration Facility

This key facility is utilised for the calibration of vacuum gauges to a secondary standard which are then used on our experimental facilities within the vacuum science laboratory and other groups at Daresbury which may require high accuracy measurements.

The Ultra-high vacuum chamber is pumped with a sputter ion pump and has numerous ports for vacuum gauges to be mounted, tested and calibrated. At the front of the system it has a small reservoir that can be filled with a calibration gas, usually high purity nitrogen which enters the chamber via a fine leak valve.

A pressure reading of the gauge under calibration is compared to another calibrated gauge which is established by a primary standard, under strict conditions from the physikalisch-Technische Bundesanstalt in Germany, which is renewed every twelve months. Each gauge has a data set of correction factors and is recorded on a certificate. The gauge head, signal and power cables and gauge controllers are tested as a set and must not be exchanged.

The calibration rig is an important facility for the vacuum science laboratory to ensure that all of the gauges used on our experimental facilities are certificated to a secondary standard, ensuring high accuracy pressure measurements and consistency.
The DC cylindrical magnetron deposition installation is designed for coating the internal surface of tubular samples. It uses an external 500 mm long solenoid with 65 mm inner bore. For the experiments a number of 38 mm internal diameter and 500 mm long stainless steel tubes with CF40 flanges at the extremes have been coated with different NEG films.

The sputter cathodes used were:

- Single metal 1mm diameter and 1m long wire (Ti, Zr, plus V or Hf)
- Two, three or four individual (single metal) 1mm diameter and 1m long wires of combinations of Ti, Zr, V and Hf twisted together, or,
- a 3mm diameter and 1m long Ti-Zr-V alloy wire with equal atomic percent of Ti, V and Zr

- Prior to deposition the system is baked for 48 hours at 250°C.
- The base and working pressure are 2×10⁻¹⁰ and 2×10⁻² mbar, respectively.
- The sputtering gas was Kr.
- Both substrate and target are sputter-cleaned prior to deposition. Deposition is carried out in DC mode and the deposition power is 60 W.
- This facility is ideal for coating tubular samples once the optimum coating conditions are known. The optimisation is best done using a planar magnetron system.
Many modern electron accelerators require an electron source from which electrons can be released via some form of stimulation. One such electron source is a photocathode, this is a material that releases electrons as a result of stimulation by light.

Here at Daresbury our electron accelerator (ALICE) uses such photocathodes as its electron source. The photocathode material of choice is Gallium Arsenide (GaAs) which is a semiconductor material that ordinarily does not emit electrons. In order to use GaAs a number of processes have to be completed before we are able to extract electrons. The surface must be cleaned to remove all contaminants, this is done using acids, atomic Hydrogen and by heating to a temperature of 580°C.

The final stage of preparation involves activation of the photocathode, this involves depositing both Caesium (Cs) and Oxygen (O₂) onto the GaAs surface which aids the release of electrons when the surface is stimulated with a laser. This activation process is measured in terms of Quantum Efficiency (QE).

The system shown is a three stage loadlock system, there are three vacuum chambers separated by a valve and each chamber has a specific purpose. The first chamber is the loading chamber where newly prepared photocathodes are inserted, the second chamber is used for atomic hydrogen cleaning of the photocathode surface. The third chamber is where the heating and activation of the photocathode takes place, this chamber can hold up to six photocathodes at any one time.

This system will be installed on ALICE in 2011, it will allow the changing of photocathodes when the machine requires and will improve performance as a result.

Some interesting facts about the system:
1) This system holds the QE record in the UK for GaAs, measured at 15%.
2) This system was designed as a result of an international collaboration between scientists in the UK and Russia.
3) This system will be installed on ALICE where the lowest vacuum pressure in the UK was measured, 6.0 x 10⁻¹² mbar, this is the equivalent of the pressure on the moon.
An experimental setup has been originally designed and built for studying the pumping and capacity properties of non-evaporable getter (NEG)-coated films. The measurement system is based on the Dynamic Expansion Method with a gas injection system that is capable of injecting the residual gases present in a typical UHV system, such as H₂, CO, CO₂ and CH₄. It allows studying planar, cup and tubular samples.

The recent modification allows measurement and investigation of electron stimulated desorption (ESD) as a function of electron energy (from 10 eV to 7 keV), of accumulated electron dose and of temperature (from -5°C to +70°C). The test system is a cylinder 0.5 m long with CF40 flanges at the extremes. Samples can be conventional materials such as stainless steel, aluminium alloys or copper, or, these same materials with different coatings to enhance their outgassing barrier or NEG properties.
Chemical vapour deposition (CVD) is a process that involves depositing a solid material from a gaseous phase. Substrate temperature is critical and can influence the reaction that will take place. The process can be carried out at atmospheric or low pressure.

Precursor gases (often diluted in carrier gases) are delivered into the reaction chamber at approximately ambient temperature. As they pass over or come into contact with a heated substrate they react or decompose forming a solid phase which are deposited onto the substrate. The films are usually a few microns thick and are generally deposited at fairly slow rates, usually of order of hundred microns per hour.

The CVD advantages are:

- uniform film deposition on the surface of complex shaped components
- coating inside narrow (less than 4 mm gap) vessels.
In a static vacuum system, thermal outgassing is the major contribution towards the total pressure, \( P \) (mbar).

\[
P = \frac{Q}{S} + P_o
\]

\( Q \) is the outgassing rate in mBar.l s\(^{-1}\)

\( S \) is the pumping speed of the pump in l/s and

\( P_o \) is the ultimate pressure of the pump.

Pumping speed of pumps, \( S \), normally vary from about 1 l/s up to around 1000 l/s, a range of 3 orders of magnitude. However, the specific outgassing rate of materials, defined as \( q = \frac{Q}{A} \) where \( A \) is the sample area, can vary typically over 10 orders of magnitude from \( 10^{-5} \) to \( 10^{-15} \) mbar·l/(s·cm\(^2\)).

In order to reduce the total pressure, \( P \), it is therefore desirable to reduce the outgassing rate, \( Q \), as much as possible. This is done through the careful selection of materials that are used in the vacuum envelope.

A system that can determine the outgassing rate of a sample is therefore of great interest to the experienced UHV expert, looking to use new materials within his vacuum chamber.

The outgassing facility based on throughput method consists of two chambers, one for the sample the other for the pumping system, separated by a small hole of known conductance. By measuring the pressure difference across the conductance plate, the sample outgassing rate can be determined:

\[
Q_{\text{Sample}} = \left( \frac{P_{\text{Samples}} - P_{\text{Pumping}}}{A_{\text{Samples}}} \right) \cdot C - Q_{\text{Blank}}
\]

Where pressures \( P_1 \) and \( P_2 \), measured in the sample chamber and the pump chamber respectively, \( C \) is conductance between these chambers and \( Q_{\text{Blank}} \) is the background outgassing rate measured without a sample.

This system benefits from a load lock, which allows samples to be loaded without the need to vent the sample and pumping chambers. This allows for a faster turnaround between samples, as the system does not require baking between samples. Also the samples are placed on a heater stage, this can either be used to determine the outgassing rates at elevated temperatures, or to determine the outgassing rate after being baked.
In a simple vacuum system the pressure defined by thermal outgassing and effective gas load is the following:

\[ P = \frac{Q}{S_{\text{eff}}} + P_0 \]

\( Q \) is the outgassing rate in mbar.l.s\(^{-1}\).
\( S_{\text{eff}} \) is the pumping speed of the pump in l/s and
\( P_0 \) is the ultimate pressure of the pump.

If a vacuum system has a long history of being under vacuum and has had many vacuum bakes then the outgassing rate of its walls \( Q_{\text{v}} \) will be reduced (so-called a memory effect). An outgassing of this vacuum chamber with a sample can be measured and compared to the measurements without a sample if both measurements were performed in the same conditions. The sample outgassing \( Q_s \) can be found if its outgassing is greater than the outgassing from the vacuum chamber, otherwise it defines an upper limit of sample outgassing.

Two techniques can be applied on this facility.

1) **Known pumping speed**
   Outgassing rate without sample measured as
   \[ Q_w (t) = P_w (t) S_{\text{eff}} \]
   Outgassing rate with a sample measured as
   \[ Q_{w+s} (t) = P_{w+s} (t) S_{\text{eff}} \]
   Subtraction of two sets of measurements gives the sample outgassing
   \[ Q_s (t) = (P_w (t) - P_{w+s} (t)) S_{\text{eff}} \]
2) Pressure rise method

In a sealed chamber of known volume $V$, the pressure $P$ rise with time $t$ as:

$$ P(t) = \frac{\int Q(t)\, dt}{V} + P(t_0) = \frac{\bar{Q}}{V} \cdot (t - t_0) $$

Where $t_0$ is time when the vacuum chamber was isolated for the pump after pumping down to UHV.

By determining the rate of pressure rise and knowing the volume of the chamber, the combined outgassing for the chamber and sample can be calculated. If a test has been preformed prior, without a sample, then a figure giving the outgassing for just the chamber, would have been obtained, which may be deducted from this value, to give a figure for just the outgassing of the sample.

An average outgassing rate without sample measured as

$$ \bar{Q}_{\text{wc}} = \frac{P_{\text{wc}}(t_1) - P_{\text{wc}}(t_{01})}{t_1 - t_{01}} V $$

An average outgassing rate with a sample measured as

$$ \bar{Q}_{\text{wc+s}} = \frac{P_{\text{wc+s}}(t_2) - P_{\text{wc+s}}(t_{02})}{t_2 - t_{02}} V $$

Subtraction of two measurements gives the sample average outgassing

$$ \bar{Q}_s = \bar{Q}_{\text{wc+s}} - \bar{Q}_{\text{wc}} $$