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Partial pressure gauges and residual gas analysis

6.1 INTRODUCTION

All pressure gauges treated in the previous chapter measure only the total pressure (or density). In general, measurement of only this total pressure does not provide sufficient information about the circumstances in a vacuum system. The total pressure, in fact, does not give any information about the presence of individual gas or vapour components and their contribution to the pressure. Nonetheless, knowledge of the partial pressures of the present gases and vapours in a wide variety of cases appears of vital importance. In view of the reliability, efficiency and the user-friendly properties of the presently available partial pressure gauges, the presence of merely a total pressure gauge can only be justifiable from the standpoint of simplicity and costs, particularly in ultra-high vacuum systems and in cases of complicated procedures carried out in vacuum.

An illustrative example of the usefulness of partial pressure measurement is constituted by a vacuum system in which the required ultimate pressure is not attained. This can have several causes. Although it may be due to leakage, the main reason is usually degassing. This degassing may, for instance, be the result of adsorbed water vapour, contamination with hydrocarbons, or simply a wrong choice of material. Finally, the required ultimate pressure can sometimes not be achieved due to malfunction of the vacuum pump. In order to find out, which of these causes play a role and what measures should be taken to reduce the ultimate pressure, knowledge of the residual gas composition is indispensable in this situation.

For processes carried out in vacuum it is often not so much the total pressure but rather the presence or absence of certain gases that are of interest. Thus, for example, in a sputter deposition process the purity of the sputter gas (usually argon) is important for the quality of the deposited thin layer. Usually, even minor impurities of nitrogen, oxygen (leakage) or hydrocarbons (pumping system) will have an appreciable effect. It is also possible that during the execution of a process, gas is released which is harmful for the process itself or for the quality of the product. A simple example is the vapour deposition

of thin layers in vacuum, where the amount of heat released by the vapour deposition source can have such high values that the degassing of the surroundings of the source increases. Even the degassing of the evaporated material itself can play a role.

There are many other practical situations which can be added to these examples, which all have in common that more detailed information on the (residual) gas composition is required. If such information is available, it is often the case that the required vacuum or vacuum process conditions can be achieved with less effort and within a shorter time. Thus, for instance, it might appear that it is preferable to use other pumping methods (e.g. with a selective high pumping speed for 'disturbing' gases), other construction materials (with lower degassing values), purer gas sources or to bake-out more carefully, etc.

The instruments developed to measure the gas composition under vacuum conditions are collectively referred to by the name *residual gas analyzers*. A residual gas analyzer (RGA) can best be described as "*a sensitive mass spectrometer whose gauge head dimensions are sufficiently small to easily be mounted at or in a vacuum chamber.*" In this sense RGA's are distinguished from other mass spectrometer systems, as for instance applied in ion implantation machines or in chemical gas analysis. The latter devices are not useful as a RGA because they are too bulky, would need long connection lines to the vacuum compartment and are too complex to be easily baked.

We note that, in cases where we deal with the analysis of a process carried out in vacuum, we could better apply the more accurate term *process gas analyzer* (PGA). Yet, also in these cases, the generally established name 'RGA' is often used.

The central part of a RGA is constituted by the mass analyzer section, where the gas particles are separated according to their mass. In this analyzer section electrical and/or magnetic fields are applied to effect this mass separation. Since these fields have no effect on neutral gas particles, these particles have to be ionized in an ion source prior to their analysis. Finally, the ions which pass through the analyzer section, are captured and detected onto an ion collector. This brings us to the basic set-up of a RGA as schematically shown in figure 6.1. By continuous variation of the field settings in the analyzer section, the ions of consecutive masses are detected at the collector, and a so-called mass spectrum is obtained. Thus, a mass spectrum of a gas mixture shows up as a series of current peaks. The partial pressures of the present gases can be derived from the heights of the various peaks. In this way, the gas composition can be determined.

In the past sixty years, several types of RGA's have been presented in the literature. We mention the magnetic deflection mass spectrometer, the cycloid mass spectrometer, the Omegatron, the Farvitron, the linear high-frequency mass spectrometer (Topatron), the time-of-flight spectrometer, the quadrupole mass spectrometer and the autoresonant trap mass spectrometer, each with their specific advantages and disadvantages. Due to the ever growing demands on ruggedness, reliability, resolution, sensitivity, scanning speed,

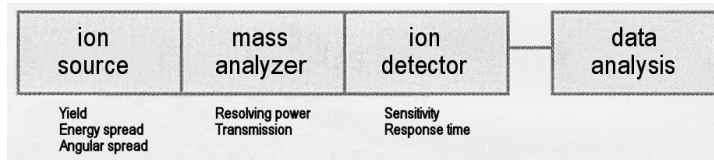


Figure 6.1

Basic design of a residual gas analyzer with quality-determining parameters.

mass range, working pressure range, operation and maintenance, of all these more or less ingenious analysis options only two instruments have developed into versatile residual gas analyzers over the years: the *quadrupole mass spectrometer* and *autoresonant trap mass spectrometer*. Within vacuum technology the *180° magnetic deflection mass spectrometer* is still applied in helium leak detectors (see chapter 8). In the following sections, these three instruments are further discussed.

6.2 THE ION SOURCE

As regards to its operation, the ion source of a RGA is comparable to a hot cathode ionization gauge. Figure 6.2 shows a classic example of a mass spectrometer source developed by Nier (1947). In an ionization chamber, a part of the present gas particles is ionized by means of electrons, which are emitted by a cathode filament and accelerated to an opposite anode. The created ions are extracted from the ionization chamber and focused through a few slit-shaped apertures into the analysis section of the spectrometer by means of an electric field. Analog to the hot cathode ionization gauge the extracted ion current i^+ can be written as

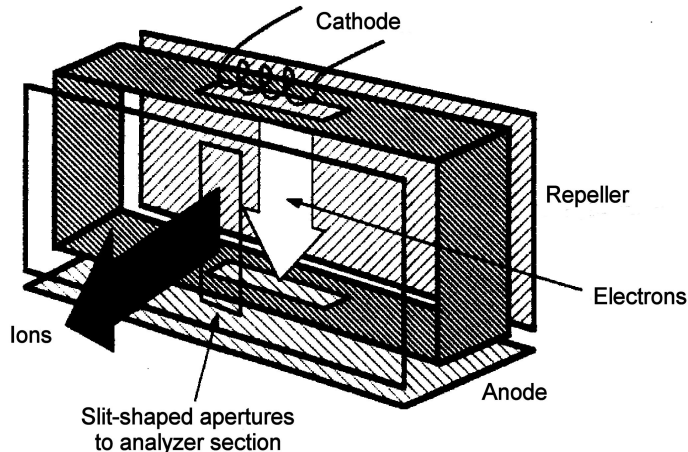


Figure 6.2

Exploded sketch of an ion source according to the Nier principle (Nier source).

$$i^+ = C p i^- \quad (6.1)$$

Where p is the ion source pressure and i^- the electron current. C is the sensitivity factor of the ion source and is determined by the gas type dependent ionization probability, the electron path length in the ion source and the extraction efficiency (= fraction of the generated ions which enters the analysis section).

As already explained in § 5.6.1, the validity of eq. (6.1) with a pressure independent C at higher pressures is limited due to the increasing influence of the secondary electrons generated by ionization. A shift of this limit to a higher pressure can be achieved by a small electron path length and consequently a small value for C . In this respect, the set-up of the *Nier source* in figure 6.2 can be considered to be suitable. After all, by choosing small dimensions for the ionization chamber, these requirements can be fulfilled simply. An additional advantage is that the emerging ion beam has a low directional and energy spread. For proper operation of some mass analyzer systems an ion beam with well-defined direction and energy is necessary.

The mentioned favourable properties of the *Nier source* unfortunately come at the expense of the extraction efficiency. In the low pressure regime the application of an ion source with a short electron path length, low extraction efficiency and therefore a low sensitivity factor imposes high demands on the transmission of the analyzer and the sensitivity of the detection system. In order to increase the ion yield from the source at a low pressure, one can increase the electron current. An excessively high electron current, however, can give rise to space charge effects in the source. This will increase the energy spread of the ions, while it possibly may also result in a further reduction of the extraction efficiency. Additional problems at low pressure are:

1. the relatively large surface area of a *Nier source*, which, moreover, is heated (thermal desorption!) by the filament,
- 2 the occurrence of electron-stimulated desorption (ESD) from the anode.

Both effects also occur to a certain extent in an *axial ion source*, but thanks to a rotation-symmetrical configuration the efficiency of this source type is significantly higher than that of a *Nier source*. Figure 6.3 shows the schematic set-up of an axial source. The cathode is positioned in the repeller hole and connected conductively. Emitted electrons are accelerated towards the anode and ionize gas particles on the way. The positive ions are axially accelerated from the ionization chamber and enter the analysis section through an extraction aperture. Axial ion sources are characterized by a robust design and a high sensitivity.

When only small (usually secluded) gas amounts are available for analysis and/or to minimize the contribution of the residual gas to the spectrum to be measured, a so-called *gas-tight ion source* can be applied. The ionization chamber in this source is connected directly to a gas admission system via a capillary tube, through which the gas to be