

Description

The present invention relates to a method of purifying a continuous flow of helium and/or neon gas; in particular, but not exclusively, the invention is concerned with the continuous production of ultra pure helium or neon (or mixtures thereof) where 'ultra pure' means that contamination with any impurity gas or gases is less than one part in ten million by volume. Helium and neon gases of this purity have many uses including as sample carrier gases in modern analytical techniques, filling gases for discharge tubes and lasers, and purge gases in the semiconductor industry.

Many processes are known for purifying continuous streams of gases. US Patent No 4,313,739 (Douglas-Hamilton) describes one process which is based on preferential ionization of contaminant molecules, and involves the steps of passing a stream of the gas to be purified along a predetermined flow path; ionising impurities in the gas; and removing the positive impurity ions from the main gas flow. In this process, the impurities are ionised by using a corona discharge to generate ions and electrons in the gas which then exchange charge with impurity molecules. Of course, this method only works where the impurity molecules have a lower ionisation potential than the molecules of the gas to be purified.

Furthermore, the efficiency of the overall process is poor, not least because the ions and electrons required to ionise the impurity are continuously removed from the gas flow by the polarising field.

It is an object of the present invention to provide an improved method for the production of ultra pure helium and neon gases on a continuous basis in a manner relatively easy to implement.

According to one aspect of the present invention, there is provided a method of purifying a continuous flow of helium or neon gas or a mixture thereof, said method involving the operations of passing a stream of the gas to be purified along a predetermined flow path, ionising impurities in the gas, and removing the positive impurity ions from the main gas flow by setting up a polarising field across said stream to cause migration of said impurity ions out of the main gas flow, characterised in that ionisation of said impurities is effected by exciting the gas stream with an electrical discharge such as to produce rare-gas metastable atoms that serve to ionise said impurities by losing energy thereto, said metastable atoms providing the predominant impurity-ionisation process.

The effectiveness of this purification method rests in the high excitation potentials of noble gas metastables. In particular, the metastable excitation potential of helium or neon is greater than the

ionisation potentials of any impurities. In addition, these metastables, being neutral, are not removed from the gas stream by the polarising field. Furthermore, the noble gas metastables readily lose their stored energy to impurity molecules they encounter by collision leaving the noble-gas as uncharged, unexcited atoms. The ionising effect of noble gas 'metastable' atoms is, of course, itself a known effect (see UK Patent Specification No 882,997) though not previously used in the purification of noble gases.

The excitation of the gas stream will generally also result in the direct production of impurity ions and also in the production of noble gas ions, which, in turn, cause ionisation of impurities. However, these latter processes are considerably less efficient at ionising impurities than by generation of metastables.

A helium/neon gas purifier embodying the invention will now be described by way of non-limiting example with reference to the accompanying diagrammatic drawing, the sole Figure of which is a longitudinal cross-section through the purifier.

Although the purifier to be described is suitable for purifying helium or neon gases individually or in a mixture, for ease of explanation the following description will be given in terms of the purification of a helium gas flow only.

As shown in the drawing, the purifier comprises a tubular member 10 defining a main gas flow path between an inlet 11 for impure helium and an outlet 12 for purified helium. The tubular member 10 is formed of a porous conducting material such as sintered metal or sintered metallized glass or quartz. The tubular member 10 constitutes a cathode of the purifier, the complementary anode 13 being formed by a metal rod positioned coaxially in the tubular cathode 10 insulating end seals 14.

A polarising field is set up between the anode 13 and cathode 10 by a DC polarising source 15. This source 15 applies a potential between the anode and cathode which is substantially less than would cause an arc or discharge in helium at the pressure existing within the purifier during operation (generally slightly above atmospheric pressure); the applied potential is, however, sufficient to cause a rapid migration of any positive ions present to the cathode 10.

In order to ionise impurities in the gas flowing through the cathode 10, a pulse source 16 is provided for supplying high frequency (100 kHz to 50MHz), high potential, short duration (less than one microsecond) pulses between the anode and cathode whereby to set up an ionising corona discharge between the electrodes. Such an arrangement is disclosed in UK Patent Specification No. 1,597,623; and will not, therefore, be further described herein except to note that since to ini-

tiate corona discharge at least one ion or electron must be present, it is prudent to provide some means for generating the required ion or electron (although background radiation could be relied upon). Consequently, the polarising source could be made adjustable to cause an initial discharge before being set back to its normal operating potential.

Surrounding the tubular cathode 10 is a cylindrical jacket 17 for collecting gas which, in operation, passes radially outwards through the porous cathode 10. This jacket 17 has an exhaust port 18 adjacent the inlet end of the main gas flow path defined by the cathode 10.

A diffuser 19 is provided in the main gas flow path adjacent to inlet 11 to establish streamline flow down the inside of the cathode 10.

In operation, impure helium typically, one part per million impurity) is supplied to the inlet 11 at a slight over-pressure relative to the surrounding atmosphere to establish a streamlined main gas flow coaxially down the inside of the cathode 10 in the direction of arrow A to the outlet 12. Due to the porous nature of the cathode 10, a small subsidiary gas flow is set up that passes radially outwards through the cathode and then in the direction of arrow B to the exhaust port 18.

The corona discharge established between the anode 13 and cathode 10 by the pulse source 16 causes ionisation of any impurity atoms or molecules within the main gas stream either directly or by first generating helium metastable atoms and helium ions which subsequently collide with and ionise any impurities. Ultra-violet radiation emitted from the transitions of the helium ions and metastables will also produce ionisation of impurities by photo-ionisation. The predominant impurity-ionising process will generally be that due to the collision of helium metastables with impurity atoms and molecules.

The polarising field set up between the anode 13 and cathode 11 by the polarising source 15 causes positive impurity ions to migrate to the cathode where they pass out of the main gas stream through the porous cathode 11 as part of the subsidiary gas flow.

The purified helium typically with an impurity level of less than one part in ten million exits the purifier through the outlet 12 while the impurities are exhausted through exhaust port 18.

The actual dimensions of the purifier will depend upon the desired flow rate of helium and the degree of purity sought. However, typical dimensions would be a tube 10 mm in internal diameter with a length of 0.3 metres.

An ion current of 4.3 microamperes would be needed to remove all of a one part per million impurity in helium flowing at one millilitre per

second at N.T.P. This assumes that all of the ion current is carried by impurity ions and that the impurities are completely removed from the main helium flow after the impurity ions encounter the cathode. In practice, ionising efficiencies of 1 to 10% are more usual.

The purifier can be thought of as analogous to a distillation column with gaseous diffusion processes tending to hinder the smooth operation of the separation. For a cathode 10 mm in internal diameter and an anode 2 mm diameter and with the flow rate and impurity level quoted in the preceding paragraph, each 5 to 10 mm section of the purifier corresponds to a plate of a distillation column and 67% of impurity will be removed in each of the sections. Thus, five sections should theoretically remove about 99% of the impurity. A typical purifier is in fact 0.3m or more in length to extend the range of purification and the maximum flow rate.

The structural and electrical characteristics of the purifier are preferably such as to avoid the development of ion densities in the purifier in the plasma region. In plasma, ions and electrons are closely associated in a gaseous cloud and the ions cannot be so readily moved to the electrodes by the applied polarising field. For a 10 mm section of purifier, with a cathode having an internal diameter of 10 mm, the polarising field current should not exceed one microampere if plasma conditions are to be avoided.

Furthermore, whilst the use of a porous cathode is a particularly convenient and efficient way of removing the impurity atoms and molecules, it would be possible to use a non-porous cathode and then to separate off from the main gas flow, the gas stream that passes directly over the cathode and within which the neutralised impurities will be concentrated.

Claims

1. A method of purifying a continuous flow of helium or neon gas or a mixture thereof, said method involving the operations of passing a stream of the gas to be purified along a pre-determined flow path, ionising impurities in the gas, and removing the positive impurity ions from the main gas flow by setting up a polarising field across said stream to cause migration of said impurity ions out of the main gas flow, characterised in that ionisation of said impurities is effected by exciting the gas stream with an electrical discharge such as to produce rare-gas metastable atoms that serve to ionise said impurities by losing energy thereto, said metastable atoms providing the predominant impurity-ionisation process.

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A diffuser 19 is provided in the main gas flow path adjacent to inlet 11 to establish streamline flow down the inside of the cathode 10.

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The corona discharge established between the anode 13 and cathode 10 by the pulse source 16 causes ionisation of any impurity atoms or molecules within the main gas stream either directly or by first generating helium metastable atoms and helium ions which subsequently collide with and ionise any impurities. Ultra-violet radiation emitted from the transitions of the helium ions and metastables will also produce ionisation of impurities by photo-ionisation. The predominant impurity-ionising process will generally be that due to the collision of helium metastables with impurity atoms and molecules.

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Revendications

1. Procédé de purification d'un écoulement continu de gaz hélium ou néon ou d'un mélange de ceux-ci, ledit procédé impliquant les opérations consistant à faire passer un courant du gaz à purifier le long d'un trajet d'écoulement prédéterminé, à ioniser les impuretés dans le gaz, et à éliminer les ions positifs d'impuretés de l'écoulement principal de gaz en établissant un champ de polarisation en travers dudit courant pour entraîner une migration desdits ions d'impuretés en dehors de l'écoulement principal de gaz, caractérisé en ce que l'ionisation desdites impuretés est effectuée en excitant le courant gazeux avec une décharge électrique de façon à produire des atomes métastables de gaz rare qui servent à ioniser lesdites impuretés en y perdant leur énergie, lesdits atomes métastables fournissant le procédé prédominant d'ionisation des impuretés.

Patentansprüche

1. Ein Verfahren zum Reinigen eines ununterbrochenen Stromes von Helium- oder Neon-Gas oder einer Mischung dieser Gase, wobei das Verfahren die Operationen des Leitens eines zu reinigenden Gasstromes längs eines vorbestimmten Strömungsweges, des Ionisierens der Verunreinigungen in dem Gas und des Entferns der positiven Verunreinigungionen aus dem Hauptgasstrom durch Erzeugen eines polarisierten Feldes quer zu dem Strom umfaßt, um eine Wanderung der Verunreinigungionen aus dem Hauptgasstrom zu verursachen, dadurch gekennzeichnet, daß die Ionisation der Verunreinigungen bewirkt wird, indem der Gasstrom mit einer derartigen elektrischen Entladung erregt wird, daß metastabile Edelgasatome erzeugt werden, welche zum Ionisieren der Verunreinigungen durch Verlieren von Energie an diese dienen, wobei die metastabilen Atome den überwiegenden Verunreinigungsionisationsprozeß schaffen.

