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AN IMPROVED APPARATUS FOR THE VACUUM DEPOSITION OF RADIOACTIVE MATERIALS

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An apparatus for the vacuum deposition of radioactive materials is described. The apparatus, an improved version of an earlier model, employes electron bombardment heating as a means of extending the temperature up to more than 3000° C.

The backing material holder can be manipulated externally allowing for the preparation of several samples during a single operation and a G-M detector combination is provided for the purpose of controlling the deposition of radioactive material.

A simple optical projection system has been developed for use when high levels of activity are to be processed.

In conclusion the operating procedure, and some of the results obtained, are discussed.

1. Introduction

In 1959 Parker described an apparatus for the vacuum deposition of radioactive materials[1]). It soon became apparent, however, that a second apparatus would be necessary in order to cope with the ever increasing demand for vacuum deposited samples for use in nuclear spectroscopy.

Once the decision had been reached to undertake the construction of such an apparatus, it was decided that the new design would incorporate those details overlooked in the earlier model, but which later became evident as experience was gained.

The principle improvements made in the new design are the following: Greater temperature range, external manipulation of the backing material holder, gamma-ray detectors, for the monitoring of radioactivity during processing, and finally, facilities for the employment of an optical projection system allowing the observation of radioactive processes at a reasonable distance from the radiation area.

The apparatus is employed mainly for the preparation of radioactive samples, there have, however, been a number of separation-depositions also carried out using the apparatus[2]).

In the following description only the working chamber is discussed since the vacuum system as a whole is essentially the same as that described in ref.[1]).

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2. Apparatus

The base plate has a diameter of 250 mm and a thickness of 15 mm. Passing through the base plate are two water cooled columns having a diameter of 12 mm and a variable length of from 5 to 250 mm. One of these columns passes through the centre of the base plate and can also be rotated. The second column is situated between that in the centre of the base plate and the base plate edge. The pumping port is on the opposite side of the base plate, as are a group of five glass to metal seals necessary for electrical connections. The general layout of the base plate can be seen from fig. 1.

2.1. HEATING ASSEMBLY

Heating of the vapour source or crucible is achieved by means of electron bombardment. The entire heating assembly passes through a port situated (indicated, but not shown in fig. 1) in the vacuum chamber wall, and when in position is in line with the axis of the first G-M detector. Details of the construction employed, and of filament

[1]) W. Parker, Nucl. Instr. and Meth. **5** (1959) 142.

[2]) W. Parker and Y. Grunditz, Nucl. Instr. and Meth. **14** (1961) 71.

placement, are shown in fig. 2 and the electrical circuit in fig. 3. The latter consists of the following components: A fully rectified d.c. power supply capable of delivering 500 mA at 3000 V negative high voltage, and a high voltage insulated (10000V) low voltage transformer rated at 25 A/12 V. Both high and low voltate supplies are controlled by means of variac controlled transformers.

As can be seen from fig. 3, the material to be heated is connected to ground potential while on side of the filament, and the filament enclosure, are connected to high voltage negative. In this manner the electrons emitted by the filament are focused by the enclosure and accelerated toward the crucible. The heating assembly is water cooled and lined on the inside of the heating area with tantalum sheet metal. For replacement of the filament, or other adjustment, the entire assembly can be removed from the vacuum chamber wall as a single unit.

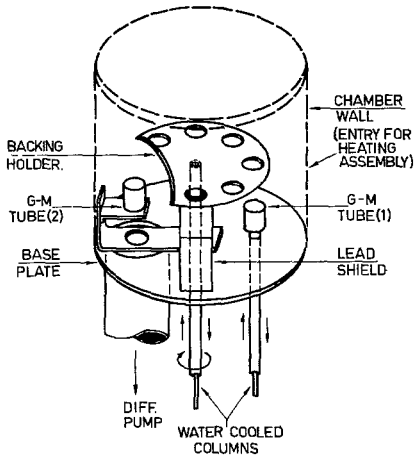

Fig 1. General layout of vacuum chamber showing the two adjustable columns, G-M detectors and backing material holder. The heating assembly (not shown in the figure) passes through the vacuum chamber wall and is positioned in between the first G-M detector and the backing material holder.

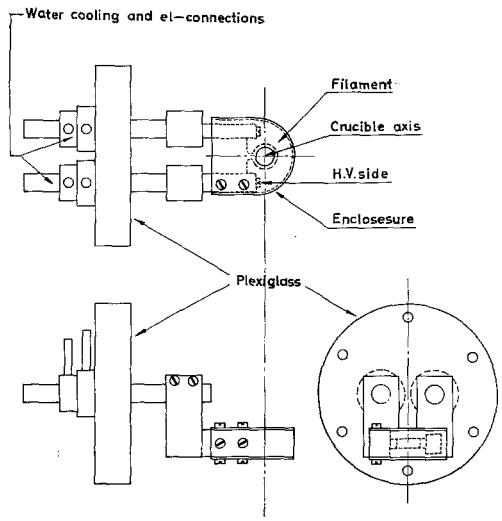

Fig. 2. Heating assembly construction showing placement of the filament (broken loop) between two massive copper supports. When in place the filament almost completely surrounds the crucible.

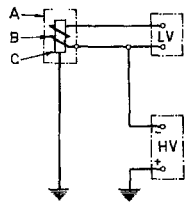

Fig. 3. Electrical circuit employed for achieving electron bombardment heating. A = filament enclosure, B = filament, C = crucible.

2.2. BACKING MATERIAL HOLDER

The backing holder consists simply of a circular plate 200 mm in diameter, with holes of suitable diameter cut around the outside edge. When in position the centre of each hole is in line with the axis of the crucible. Thus, one or more samples can be prepared by turning a new backing material into position. The holder is fixed to the centre column and can be raised and lowered as well as rotated. Water cooling of the backing material is not made use of with the arrangement described.

2.3. DETECTOR SYSTEM

For the purpose of monitoring the preparation process, two commercial G-M gamma-ray detectors are employed. One of these detectors is positioned under the heating assembly, fig. 1, and fastened to

the top of the first column. The second detector is mounted on a special bracket situated directly over the pumping port. The two detectors are shielded from each other by a lead wall which effectively divides the base plate in two halves. The high voltage connections for the two detectors are taken out through the base plate via two of the glass to metal seals provided.

As mentioned above the two detectors employed are only sensitive for gamma-radiation. However, in the event of the apparatus been used for work with pure alpha- or beta-ray emitters, thin window counters may be substituted. The construction of such a detector, having the overall dimensions of 30 mm diameter, 22 mm high and a window diameter of 7 mm, has recently been described by Andreen *et al.*[3]), and should prove very suitable for this application.

2.4. OPTICAL VIEWING SYSTEM

The optical viewing system is really simplicity itself, but should prove to be of considerable value when high levels of activity are to be processed.

In the present instance the authors were fortunate enough to have a projection lens in their possesion it being only necessary to mount this lens on a special bracket made so as to correct for a too long focul length, which in turn could be secured over one of the visual observation ports in the vacuum chamber wall. When in operation a considerably enlarged image is projected, from the inside of the vacuum chamber, of the heating assembly and crucible. Prior to, and at the conclusion of, operation other means of illumination can be employed.

2.5. OPERATION OF THE APPARATUS

No difficulty has been experienced in the operation of the apparatus. Degassing of the crucible takes place by heating only with the filament and with the backing material turned out of position. No water cooling of the backing material is employed, instead the backing material is placed in intimate contact with the polished surface of an aluminium rod previously treated with a thin layer of vacuum grease. The dimensions of the rod are 25 mm diameter and 25 mm high. This arrangement has proved to be perfectly satisfactory.

After degassing the filament temperature is decreased and the backing moved into position and adjusted to the correct height above the crucible. The activity of the crucible is measured by raising the first detector to within 5 mm of the crucible bottom. After the measurement the detector is lowered again and the filament temperature increased up to the temperature of electron emission.

The high voltage supply is switched on and the high voltage increased until the desired temperature is reached (2000° C is reached in about 30 sec), held at this temperature for a 60 sec period, and the power turned off again. The backing material holder can now be rotated 180° bringing it over the second G-M detector in which position any activity present can be measured and compared with a second measurement of the crucible.

The vapour sources or crucibles used in the apparatus are the same as those described in ref.[1]).

3. Discussion

The described apparatus has been in operation for a two year period and has presented no serious difficulties during that time. The preparation of radioactive samples has been greatly facillitated by the incorporation of the external manipulation controls, and also, by the improved heating system.

As has been pointed out previously[4, 5]), vacuum deposition affords a means of preparing thin uniform deposits on thin foils and films providing that a reasonable degree of efficiency can be achieved.

In the present instance this has certainly proved to be the case.

The results of the separation-deposition work carried out using the apparatus described above

[3]) C-J. Andreen, W. Parker and H. Slätis, Nucl. Instr. and Meth. **15** (1962) 205.

[4]) W. Parker, M. De Croes and K. Sevier, Nucl. Instr. and Meth. **7** (1960) 22.

[5]) W. Parker and H. Slätis, Sample and Window Technique, To be published in: Alpha, Beta and Gamma-ray Spectroscopy. Editor: K. Siegbahn (North-Holland Publishing Co., Amsterdam, 1963).

have been reported in ref.[2]), and will not be dealt with here. Recently, however, DeVoe and Meinke[6]) have completed a study of this particular technique the results of which indicate the methods application to the rapid separation of radioactive materials.

Acknowledgement

The authors wish to express their gratitude to Professor Kai Siegbahn for his interest and support during the development and construction of the apparatus.

[6]) J. DeVoe and W. W. Meinke, Analytical Chemistry (in print).