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DEPOSITION BY ELECTRON BOMBARDMENT AND WEIGHING UNDER VACUUM OF THIN HIGH PURITY BORON LAYERS

by

J. VAN AUDENHOVE H.L. ESCHBACH and H. MORET

1964

Joint Nuclear Research Centre
Geel Establishment (Belgium)
Central Nuclear Measurements Bureau (C.N.M.B.)

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Received 30 April 1963

The equipment and the techniques for the deposition of very uniform thin boron layers by electron beam evaporation are described. As substrates aluminium or quartz discs with a diameter of 48 mm and a thickness of 0.2 mm were used. The experiences with a specially designed vacuum micro-balance are discussed. Boron layers of 50 $\mu g/cm^2$ could be determined by weighing to within $\pm$ 4 μg which is equivalent to $\pm$ 0.2 $\mu g/cm^2$ for this diameter.

1. Introduction

An experiment to measure with high accuracy the fission cross section of fissile material required a reference to be used in the counting technique. It was decided to take boron as the reference material, which has to be deposited as a thin layer on a quartz or pure aluminium disc. The total amount of ^{10}B must be known with the best accuracy presently achievable.

This means that the mass of the layer must be measured, the isotopic composition must be known, the chemical purity must be high, and the impurities must be analysed both qualitatively and quantitatively. The base material for the preparation of the reference is natural boron of high chemical purity.

As the preparation technique vacuum evaporation was chosen, this being the only method to render very pure metallic boron deposites. However, the high temperatures required (2500° C) and the high purity asked for rule out the use of resistance heated crucibles. We, therefore, evaporated the boron by electron bombardment heating in an experimental set-up in which no molten boron comes into contact with any crucible material. The particular type of electron gun developed is of a very simple construction and showed to give very uniform layers.

The total amount of deposit is weighed in situ, i.e. in the vacuum evaporation apparatus, to avoid inaccuracies resulting from handling, oxidation, and ad- and desorption effects. A vacuum balance was specially designed for this purpose.

The chemical analysis which is extremely difficult as far as traces of carbon are concerned will not be treated here. The isotopic analysis does not seem to meet specific difficulties, and fractionation effects during evaporation are expected to be negligible but this part of the investigation is not yet finished.

2. The Production of Thin Boron Layers

2.1. THE VACUUM EQUIPMENT

Fig. 1 shows a schematic diagram of the vacuum system. The high vacuum chamber (1) is a cylindrical stainless steel tank having a height of 600 mm and a diameter of 500 mm. Both, base and

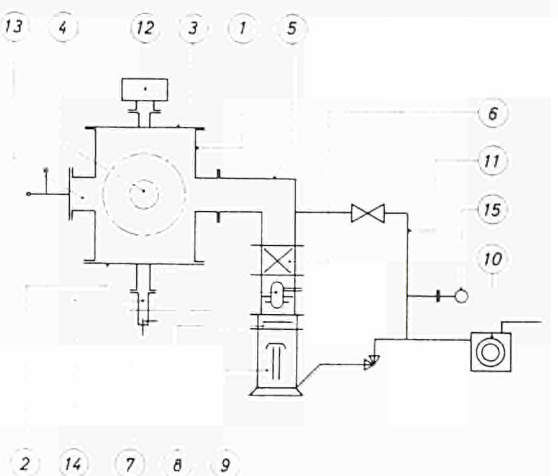

Fig. 1. Schematic diagram of the vacuum system.

top cover (2,3) of the tank can be lifted and lowered by means of a gallows in order to give full access to the inner chamber. A door (4) with a lead glass viewing port allows to charge the crucible, to exchange the electron gun and to watch the evaporation system during work. The complete vacuum tank, the top and the base cover, as well as the elbow (5) are double walled, so that they can be cooled or heated by cold or hot water respectively.

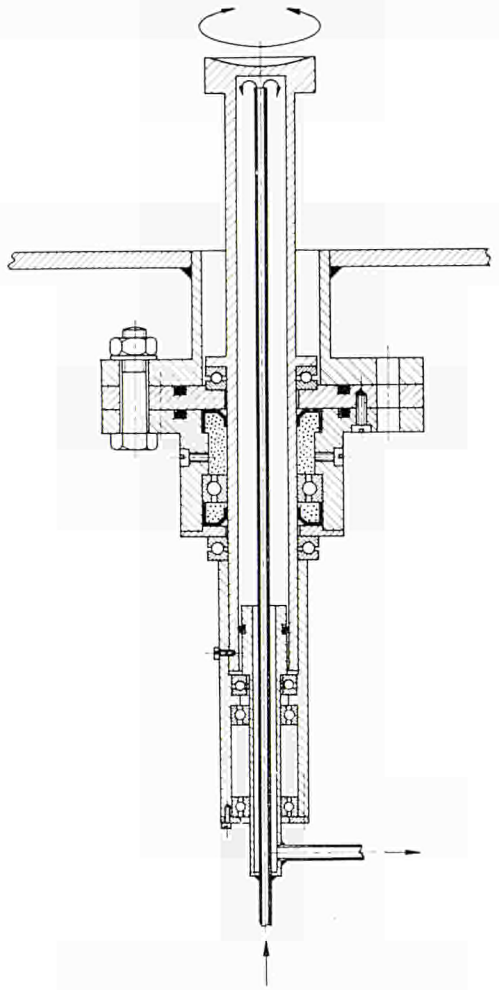

Fig. 2. Water-cooled copper-support.

The pumping duct is 250 mm in diameter and contains an elbow (5) a high vacuum valve (6) a liquid nitrogen trap (7) and a watercooled baffle (8). A 2000 l/sec oil diffusion pump (9) backed by a 25 m^3/hr rotary pump (10) and a bypass (11) complete the pumping line.

All high vacuum seals were made with Viton "O" rings or with silicon gaskets of trapezoidal cross section. The high vacuum pressures are measured with a high speed ionization gauge. A final vacuum of 10^{-6} torr can be reached.

2.2. THE EVAPORATION EQUIPMENT

For reasons of simplicity and in order to have an electron system of small dimensions it was decided to use work accelerated electron guns. First tests were made with a hairpin cathode. But due to the very low thermal conductivity of boron the narrow electron beam would rather drill a hole into the boron than melt it to a uniform and steady pool.

To overcome these difficulties we used:

1. a water-cooled copper support which can be rotated, fig. 2,

2. a ring cathode (fig. 3a) (1) connected to a combined linear and rotary feed-through (2).

The electron gun (fig. 3b) is made from 0.3 mm tantalum sheet, the cathode (A) is a 0.7 mm ϕ tantalum wire forming a loop of 40 mm in diameter. First experiments with a rather open gun structure showed that a high percentage of the total electron current left the gun through its top opening. By inserting the cylinder (B) these electron losses could be eliminated and at the same time the focussing could be improved.

The top of fig. 3a gives a schematic illustration of the substrate mounting and shielding. In most experiments the boron layers were deposited on quartz or aluminium discs (3) (48 mm in diameter, 0.2 mm thick) which were held by 3 Ta- wire hooks. The connection to the balance was made by a thin quartz wire (4). A set of shielding screens, two flat ones (5, 6) and two concentric tubes (7, 8) prevents the deposition of scattered boron atoms on the back of the substrate. All shielding screens are in good thermal contact with the top cover. During weighing the bottom opening of tube (7) is closed by a metal plate (9). Thus faulty measurements due to radiometer effects are avoided.

2.3. THE EVAPORATION PROCEDURE

Typical working conditions for a gun shown in fig. 3 are:

acceleration voltage	10 kV,
electron current	100 mA,

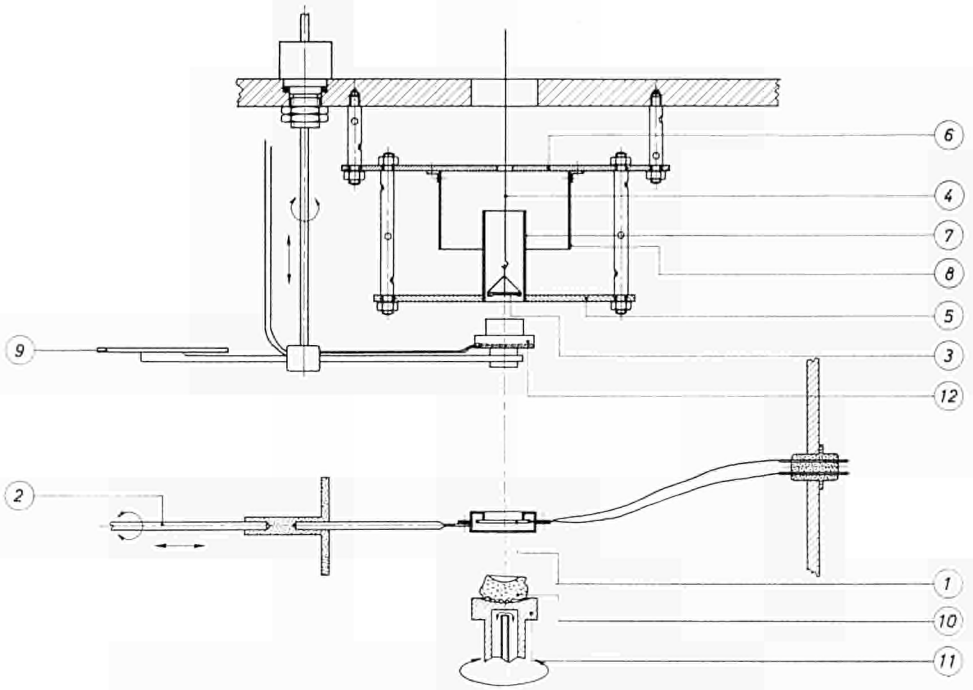

Fig. 3a. Evaporation arrangement and substrate mounting.

spot size 12, . . ., 15 mm,
evaporation rate 5 μg min^{-1} cm^{-2},
distance filament-boron crucible 30, . . ., 35 mm,
distance boron crucible-substrate 350 mm.

Even when using electron beam evaporation techniques great care has to be taken that no impurities from crucible materials are introduced into the deposit. Therefore, an arrangement was chosen (fig. 3a) in which the boron formed its own crucible (10) which was placed upon the water-cooled copper support (11). As starting material small pieces of crystalline boron were used. In a series of melting operations these were transformed into a solid crucible of about 20 mm in height and 20 mm in diameter. The very low thermal conductivity of boron allows to maintain a high temperature gradient across the crucible and no molten boron gets into direct contact with the cooper support.

Before a definite boron evaporation starts the substrate disc is outgassed at 400° C by bringing it into contact with a small oven (12) which can be inserted into the bottom opening of tube (7) in fig. 3a, and which can be moved by a suitable feedthrough. In order to get a steady, melting boron pool in the crucible (which is essential for the production of uniform layers free from pinholes and projected boron particles) the boron is melted and outgassed for five minutes with an electron beam power which is about 20% higher than that used for the evaporation itself. After this the power is decreased and the screen shielding the substrate is turned away.

3. The Vacuum Balance

3.1. GENERAL CONSIDERATIONS

The obvious way of measuring layer thickness is to weigh the substrate before and after the evaporation cycle with a normal high precision micro-

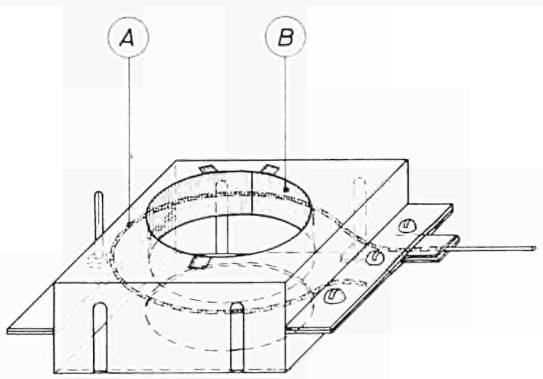

Fig. 3b. Electron gun.

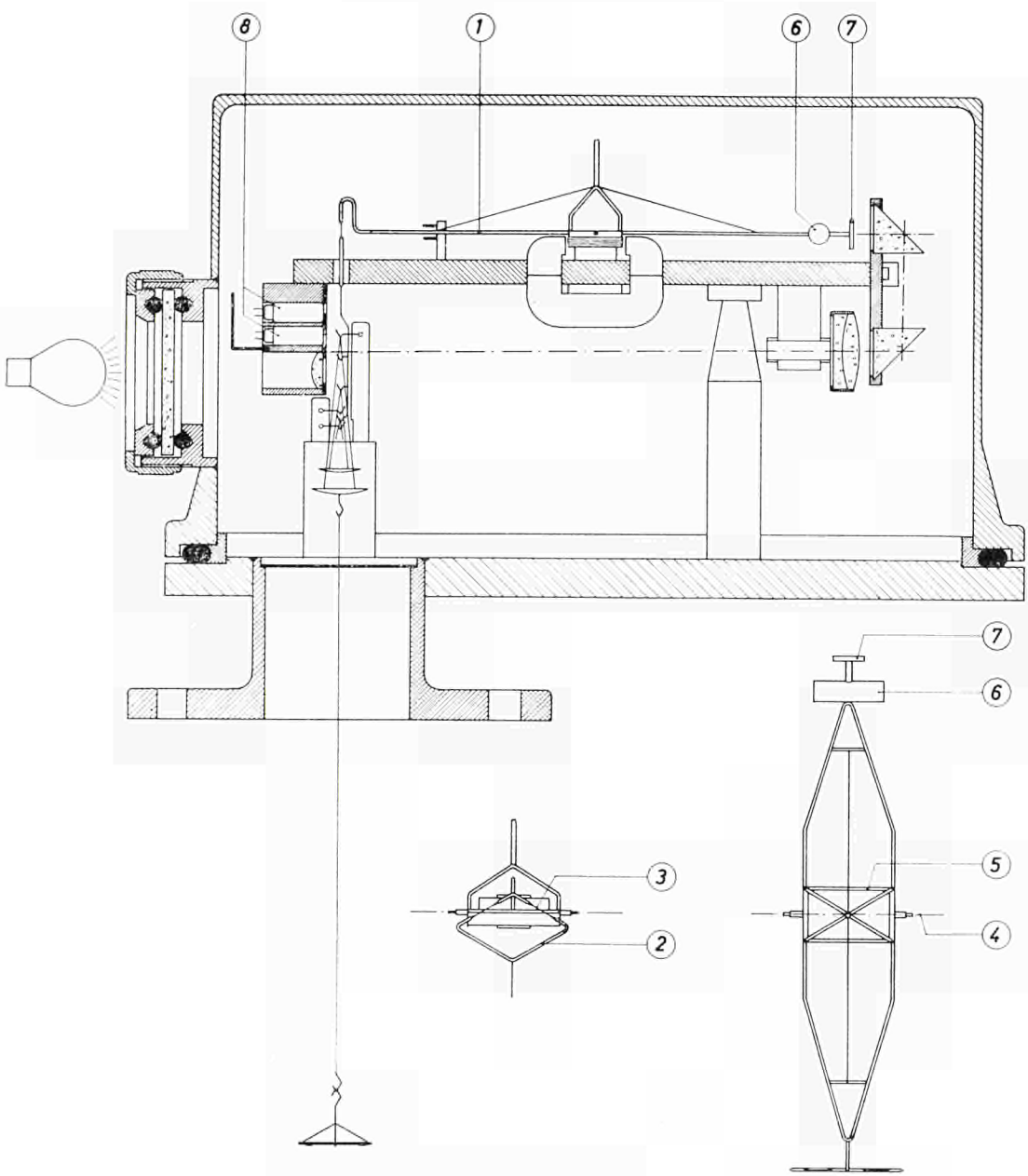

Fig. 4. Vacuum balance.

balance. However, experiments indicated that the mass of a substrate shows unpredictable changes when it has been in high vacuum and/or subjected to baking out. These mass variations seem to depend largely on the material (e.g. very pure aluminium behaves much better than quartz) and on the way the surface of the substrate has been treated. Mass variations between $\pm$ 6 μg for pure Al and $\pm$ 50 μg or more for quartz plates of a total surface area of 40 cm^2 have been found. Most probably, these variations are connected with ad- and desorption of water layers, evaporation of solvents, handling damages etc. Besides, oxydation and uncertainties in the degree of oxydation prevent the total amount of pure material to be established with accuracy.

For these reasons we decided to weigh the sample *in vacuo* (no oxydation) and in the position in

which the evaporation takes place (check on baking-out process, desorption effects, etc.).

A summary of the design requirements we set for the vacuum balance looks as follows:

1. maximum charge of 2 g, combined with an accuracy of $\pm$ 1 μg;

2. check on zero point of the balance, or absolute stability of zero point to within $\pm$ 1 μg;

3. electrical in operation for the mass changes expected;

4. dependable in operation, even under the rather arduous conditions (vibrations!) of a vacuum apparatus.

A few balances suitable for operation under vacuum are available on the market, even with a load of 1 g and a claimed accuracy of $\pm$ 1 μg. But none of these meets all 4 requirements, notably nrs 2 and 4.

3.2. DESIGN OF THE BALANCE

The balance (fig. 4) we developed for our purpose has the following features:

1. The quartz beam (1) is covered with an Al layer (to prevent electrostatical charging).

2. An all-quartz load suspension construction (2) is welded to the beam via inclined quartz torsion fibres (3). So the pivot point cannot shift.

3. The main suspension wires (4) are tungsten wires, 50 microns thick, welded to Mo ribbons which are welded to the quartz beam.

4. The center part of the beam is a quartz frame (5) on which a coil of copper wire is wound. This coil is placed in the uniform field of a permanent magnet. The tungsten suspension wires conduct the current through the coil.

5. One end of the beam carries a compensating weight (6) (a quartz rod) and a plane mirror (7).

6. The balance point of the beam is detected by a collimator equipped with photo-resistors (8). The parallel beam of light coming from the collimator is reflected by the mirror on the balance beam into the collimator.

7. The use of photo-resistors makes the electrical circuitry very simple (fig. 5). The output of the amplifier is fed back to the balance coil and the coil current is recorded.

8. A mechanical change-over system enables the operator to replace the sample by a calibrated weight. A third position of this mechanism places an extra weight of ca. 1 mg on the balance in order to measure the electrical calibration factor of the balance. The accuracy of this balance is $\pm$ 1 μg independent of slow changes in the position of the balance.

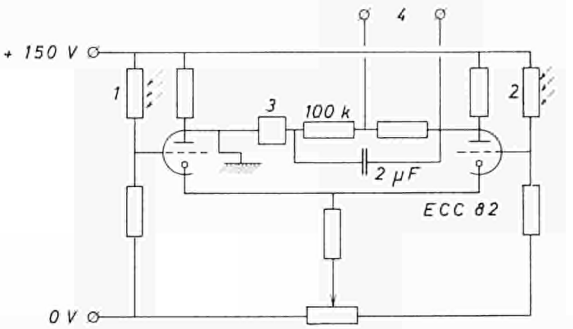

Fig. 5. Electrical circuit. 1, 2. photoresistors; 3. balance coil; 4. output to recorder.

3.3. WEIGHING PROCEDURE

After mounting the thoroughly cleaned disc and after evacuating the system a mass determination is done. Then the substrate is heated for 30 to 60 minutes. After cooling a new mass reading is done if necessary. This treatment is repeated (see below). Then the boron is evaporated onto the support. After this another measurement is done. From this measurement and the last one before the evaporation the total mass of boron layer is derived.

4. Results

4.1. UNIFORMITY OF THE LAYERS

In a separate test run the uniformity of the deposited boron layers was controlled. For these experiments the vacuum balance was not needed. Optically flat glass plates were mounted at a distance of 350 mm from the boron crucible. They could be heated to 400° C and were kept at 350 to 300° C during evaporation. This gave pinhole-free boron layers the uniformity of which was measured photometrically. Over an area of 50 mm in diameter the layers were uniform within $\pm$ 0.6% and for an area of 72 mm in diameter the uniformity varied within $\pm$ 1.7%.

4.2. MASS DETERMINATION

In a separate experiment it was ascertained that the baking-out cycle brings the support to a higher temperature than that during the evaporation cycle. Thus we may be sure that no mass change of the support itself occurs between the mass readings before and after evaporation. Normally, after the first heating to 400°C for one hour, the mass of the support is stable. This is checked by repeating the baking-out cycle 2 or 3 times. Then the mass readings lie well within a $\pm$ 1 μg interval.

After the evaporation a slow increase of the mass reading can be observed:

a) over ca. 5 μg during the first 30 minutes after the evaporation was stopped; this effect is due to radiometer effects;

b) over ca. 5 μg during the following 12 hours (after this the mass seems to be stable). This very slow increase is still unexplained. It might be related to adsorption effects.

Further study is required before it can be stated that all effects are clear and no systematic errors influence the results.

Nevertheless, the end reading may be assumed to be correct to within $\pm$ 3 μg the mass variation sub b) included. So the maximum error is $\pm$ 4 μg which is equivalent to $\pm$ 0.2 $\mu g/cm^2$ for the surface area of our supports. The layer thickness being ca. 50 $\mu g/cm^2$ the maximum relative error is $\pm$ 0.4%.

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