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NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH
Section A

Computer-controlled preparation of thin tungsten layers

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Abstract

Tungsten layers of 1–6 mg/cm^2 with diameters of 10 mm are needed as heavy-ion targets and as flat or domed windows for in-beam ion sources. They are prepared by a high-vacuum evaporation–deposition process carried out in a computer-controlled apparatus. The tungsten, placed in the water-cooled crucible of an electron-beam gun, is gradually heated and evaporates at about its melting point ($T_m = 3643$ K). The tungsten is deposited onto 9 mg/cm^2 copper backings which are preheated by the thermal radiation from the evaporant. The deposition rate is controlled by a quartz crystal monitor. After the high-vacuum deposition process, the copper backings are removed by selective etching and the self-supported tungsten foils are characterized by their areal mass. The features of the commercially available evaporation unit used are discussed by way of this deposition process.

Keywords: Thin films; Self-supported foils; Tungsten

1. Introduction

With $T_m = 3643$ K, tungsten has the highest melting point of all the metals. Self-supported tungsten layers are needed as heavy-ion targets and as flat or domed windows for in-beam ion sources [1]. For the successful preparation of tungsten layers, one must find a suitable backing material which when removed, leaves an unstressed tungsten film, an etching solution which etches the backing material much better than tungsten, a deposition process which avoids any impurities, and a good, controllable vaporization process.

2. The evaporation unit

A computer-controlled, commercially available evaporation unit is used for the preparation of thin tungsten layers. The high-vacuum evaporation unit, a Vanguard LH 470®, which was constructed by Balzers Process Systems, consists of a main chamber and a load lock with a separate pumping unit. For reasons discussed later, the process is run without utilizing the load lock. In the main chamber, there are provisions for two thermal heaters with a power of 2 kW, a glow-discharge cathode with 2 kV DC and 1 A maximum, an electron-beam gun with 6 kW power and a water-cooled, sliding copper crucible. The different heating systems are arranged at the bottom of the stainless-steel chamber. The eight-position substrate turntable is situated at the top of the chamber opposite the heaters. The quartz crystal thickness monitors, located at two different positions near the turntable, are used for the measurement of deposition rate and thickness.

The complete instrumentation; i.e., pumps, vacuum valve control, turntable, crystal monitors, doors, load lock pass through and load robot, and electrical control of the heating systems, is managed by a Siemens process computer. This process computer sends all the available data to a personal computer, which is the operator interface, and utilizes a graphics-oriented windows-based software. The main level shows all the information about the current process in numerical or graphic form. Further, the software levels allow the design of a complete deposition process or any part of it. Because the procedures and process data are saved for reuse by the computer, the deposition process no longer depends on the feelings of the operator. A typical data output is shown with the process description. The electron-beam gun as well as the thermal heaters can also be operated manually, which is quite important for preheating the material, and especially for designing new processes and finding suitable process parameters.

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3. The preparation

Copper foil, 10 cm × 15 cm × 9 mg/cm^2 (99.9%), is the backing for the tungsten layers. The foils are preheated by resistance heating until they are red-hot. This preheating produces unstressed backing material. After the preheating, the foils are formed depending upon the purpose of the future tungsten layer. The standard dome-forming tool has a diameter of 10 mm, so there are multiple formed areas on a single backing foil. The foil is then mounted in a holder about 15 cm above the crucible of the electron-beam gun. This distance is necessary to ensure that the backing has reached the proper temperature before the deposition starts. This construction makes a load-lock transfer impossible, because the holder is too large for the pass through. Fig. 1 shows the backing mounted above the electron-beam gun.

For better evaporation of tungsten, the material is preheated (with the backing rotated out of the way) to partially melt it and form a convex surface. This allows a steady evaporation rate during the deposition process. After this preheating, the substrate is rotated into a position above the crucible using the turntable.

The different parameters for the electron-beam gun as well as for the complete deposition process controlled by the deposition rate i.e., the thickness, are entered into the computer in advance so that the process can run automatically. Nevertheless, it is possible to vary the parameters of the electron-beam gun during the process.

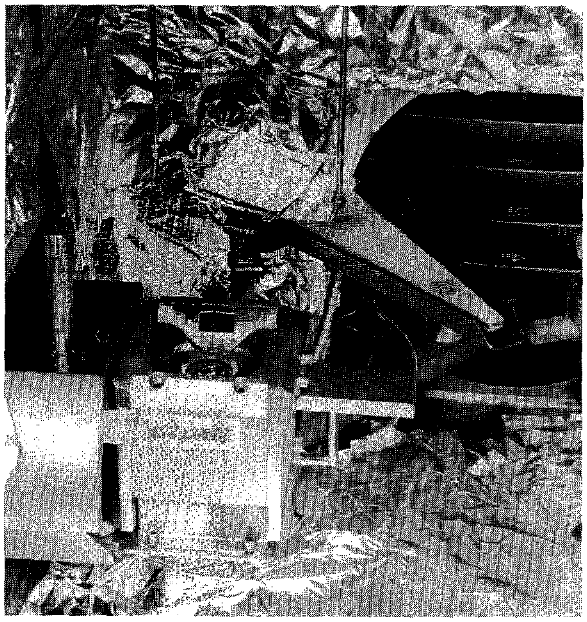

Fig. 1. Copper backing situated above the electron-beam gun.

During the soak-and-rise phase, the thermal radiation of the evaporant is used to heat the backing. Sufficient heating of the copper foil is necessary for the successful removal of the tungsten layers. The temperature of the copper foil must be above 970 K. To accomplish this

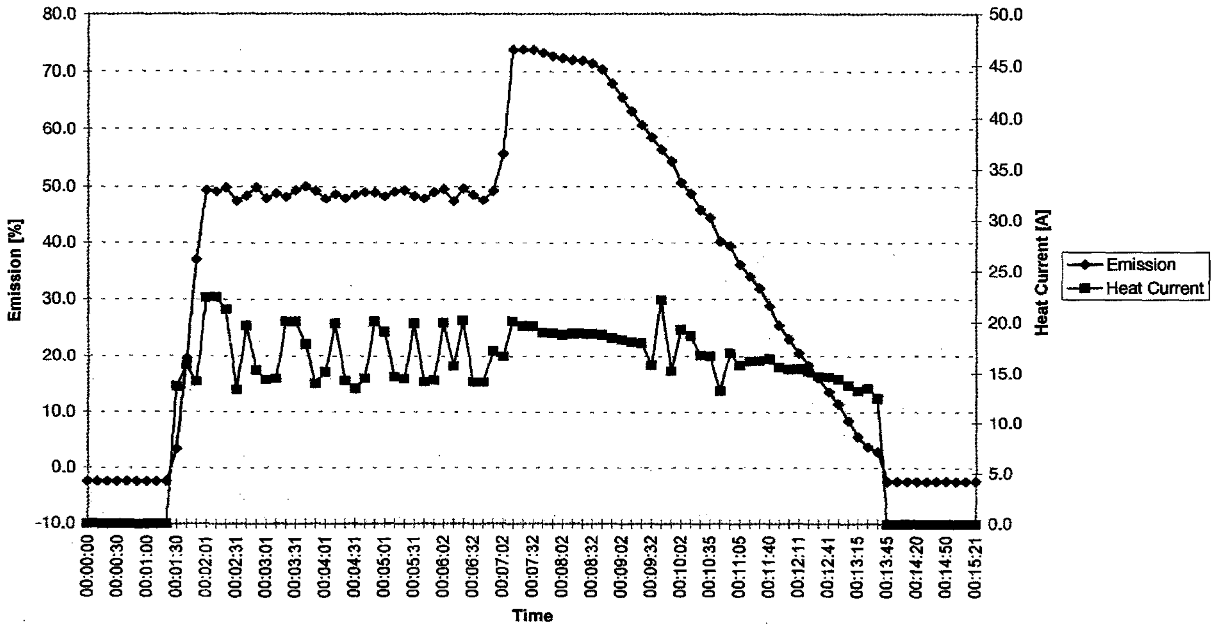

Fig. 2. Emission (%) and heat current (A) during the deposition processes.

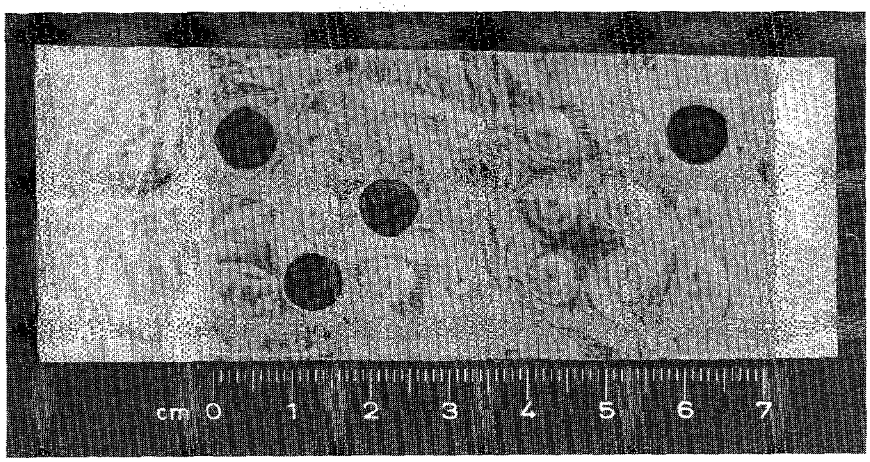

Fig. 3. Tungsten-coated copper backing, several targets already removed.

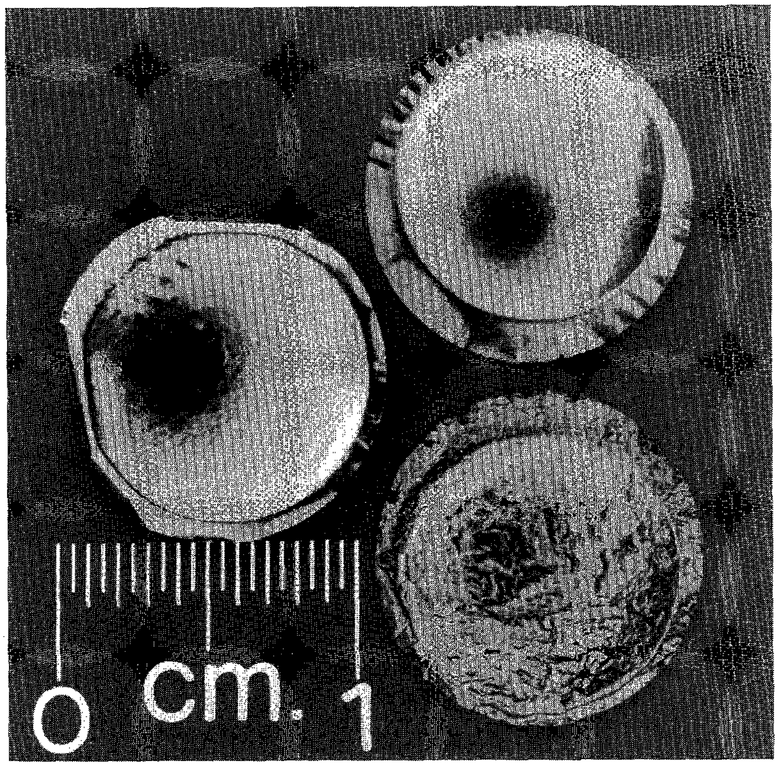

Fig. 4. Tungsten targets.

necessary heating of the backing, we have adopted a first-soak phase of 5 min at 40–50% power followed by a fast step of the second-soak just below the deposition power (approx. 75%) for half a min. Fig. 2 shows the power as a percentage of the emission current during the different phases and the heat (filament) current. Because of the short rise and second-soak time and the second-soak temperature being so close to the evaporation temperature (necessary for this particular deposition), the figure does not show a well-defined second-soak. After the deposition and a reduction of the heating over several minutes, the deposition process is complete. The tungsten on the copper foil stays in the apparatus for several hours, for further cooling.

The targets are cut out of the foil with a punch. In Fig. 3, a coated backing is shown where several targets have already been removed. The copper backing is removed by selective etching with 30% nitric acid. After 5 min in the acid, the targets are cleaned in distilled water and removed with a target frame. The tungsten targets dry on the target frame until they fall off. Self-supporting tungsten layers of 1–6 mg/cm^2 with diameters of 110 mm can be obtained. Some examples are shown in Fig. 4.

4. Discussion and outlook

The quality of the tungsten targets depends mainly on the quality of the backing. As shown in Fig. 4, the structure of the backing is copied by the tungsten layer. Also, if the backing is too cool during the deposition process, the tungsten layers crack immediately when put into the acid etch. At higher preheating temperatures, but still below 700°C, the tungsten layers roll up in a tight coil after etching and cannot be used. For a detailed analysis of the behavior of tungsten layers produced at different temperatures, we plan to install a thermocouple-based temperature measurement system. However, due to the large thermal mass of the thermocouple and its insulation compared to the thin copper foil, accurate temperature measurements will be difficult to obtain. At the same time, we plan to install active substrate heating, so that the backing does not have to be heated by the radiation from the evaporant. This should make the process easier to control. The backing holder can be constructed differently, to allow loading through the load lock. Substrate loading and unloading without opening the chamber door will allow shorter process times and higher vacuum and therefore, higher quality targets.

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References

[1] R. Kirchner, K.H. Burkhard, W. Hüller, O. Klepper, Nucl. Instr. and Meth. 186 (1981) 295.