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Knudsen Type Vacuum Evaporation Source

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ABSTRACT

Various types of resistively-heated evaporation sources are used for the vacuum deposition of materials in thin film form. However, the life of such boats or crucibles is short. This paper describes a versatile crucible for Knudsen type effusion process of evaporation for the preparation of thin films of sublimating materials, which have high vapour pressures at temperatures below 1000 °C. One representative micrograph of a thin film of *CdSe* obtained by vacuum evaporation from this crucible is also reported.

1. INTRODUCTION

Thin film deposition by vacuum evaporation is being widely used in the study of the properties of thin films, metallisation of contacts on devices, development of electronic devices by growing good semiconducting films and a host of other applications. Resistive heating is the most commonly used method for vacuum evaporation, as it is simple and cost-effective. Generally[1] the material is evaporated from a resistively-heated filament or boat made of some refractory material in a suitable configuration. However, the life of resistively-heated boats is short and in normal practice the boat is discarded after a few runs, because either it is corroded or it becomes brittle to handle further for the next charge. Moreover, it is difficult to control the rate of deposition in such boats.

The materials used for the deposition of thin films are in the form of powder, wire, sheet or crystalline lumps. The use of filaments or boats contaminates the evaporant due to alloying and hence the thin film also. Dubey[2] has observed the presence of molybdenum in vacuum-evaporated *Ge* films, when the evaporation was performed from a molybdenum boat. Also the microstructure and hence the physical properties of the condensed thin film and its thickness profile depend on the material and the shape of the filament used for vacuum evaporation. This becomes pertinent when alloys or compounds which decompose are evaporated.

An ideal evaporation source should yield the cosine distribution. But such an ideal Knudsen[3] source is not possible in practice for routine evaporation work. In the literature[1,4] various filaments have been described, but their profile of evaporation is not truely cosine, as expected of a point source. For materials which sublimate, the problems are many. Chopra[4] has described a multiple Knudsen source, which gives a uniform deposit due to the superimposition of different beams. Drumheller[5] has described another useful source for sublimating materials. However, the ideal source of evaporation is the Knudsen cell, widely used in molecular beam epitaxy. This source is a point source for all practical purposes. It has a long life and is less cumbersome to work with, but is costly.

The present paper describes a resistively-heated source with the advantages of a Knudsen source and cost-effectiveness. It has been found suitable for vacuum evaporation of almost all sublimating materials up to 1000 °C. In particular, the source has been found convenient for the preparation of vacuum-evaporated thin films for transmission electron microscopy for the study of the various stages of growth on different types of substrates at room temperature and elevated

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temperatures under different rates of evaporation. Over the years, this crucible[6,7] has been used successfully for the vacuum evaporation of a large number of materials, like *CdSe, CdTe, CdS, ZnSe, InSb, Bi, Sb, As, Ga, In, Se,* and *Te*. One micrograph of the microstructure of a thin film of *CdSe* prepared by effusion from this crucible is also reported in this paper.

2. EXPERIMENTAL DETAILS

For the routine laboratory evaporation a very useful, durable and easy to assemble Knudsen type source was used, which is shown in Fig. 1. It was made by machining from a good quality rod of steatite about 2 cm in diameter and its subsequent sintering by baking for 24 hr at 1000–1100 °C in an oven. The tungsten heating coil was prepared by winding around a specially prepared jig and was cleaned by heating in *NaOH* solution for some time. The freshly prepared crucible and the heating coil were thoroughly baked in vacuum for 10 min at 1000–1100 °C, before using the crucible to evaporate the material. The evaporant materials were evaporated at temperatures of the crucible not exceeding 1000 °C.

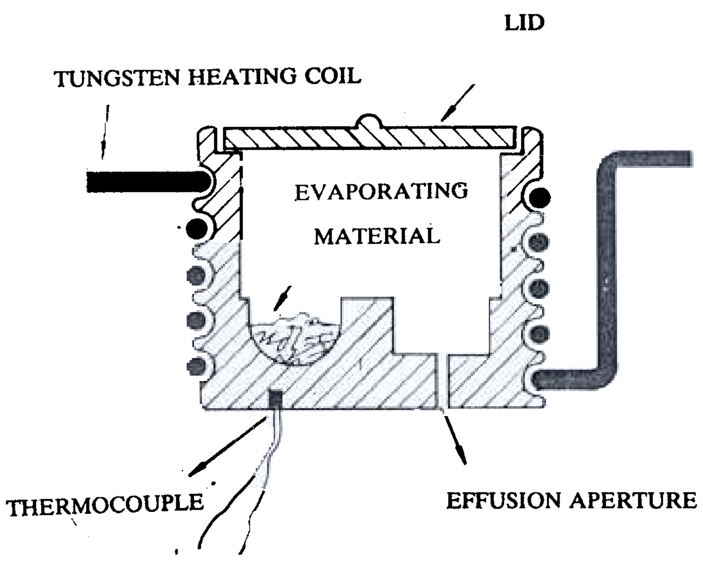

Figure 1. Vacuum evaporation crucible.

The rate of evaporation was varied by changing the temperature of the crucible by regulating current in the tungsten spiral encircling the crucible. Some salient features of the vacuum assembly are given below.

A substrate heater made of stainless steel and placed inside the vacuum system was employed. The substrate heater had a 20 W Kanthal heating element so that the temperature of the substrate could be regulated from outside the vacuum system from room temperature to about 700 °C. The substrate temperature was measured by a copper-constantan thermocouple incorporated in the substrate heater. The temperature of the substrate heater could be regulated by varying the current passing through it. The temperature of the substrate was kept constant by keeping it in thermal contact with the substrate heater. Evaporation was started when the temperature of the substrate had remained constant for about half an hour and the heating current was switched off as soon as the evaporation was over.

An aluminium shutter was interposed between the crucible and the substrate. This shutter could be operated mechanically from outside the vacuum chamber. The shutter was kept closed till the crucible had attained the required temperature and was opened only for the duration of evaporation.

In the present setup, the rates of evaporation could be varied from 10 to 200 Å/s and the substrate temperatures from room temperature to 700 °C. The films obtained were in the nucleation, coalescence and continuous stages up to 1500 Å thickness. Good quality films have been obtained on the substrates: (i) air-cleaved clear quality mica, (ii) air-cleaved *KCl* crystals, (iii) highly-doped *KCl* crystals, (iv) amorphous carbon films, and (v) glass.

3. RESULTS AND DISCUSSION

This Knudsen type source has been used conveniently and routinely by the author for vacuum evaporation of sublimating materials. The important precaution that has to be observed is that the temperature of the crucible should not be allowed to exceed 1000 °C. Beyond this temperature, steatite alloys with the tungsten heating spiral and for higher temperatures different crucible materials are required. Also the distance of the aperture from the substrate should be large to obtain thin films of uniform thickness. The distance has to be kept large to avoid direct heating of the substrate due to radiation from the heating coil. The interposed shutter wards off this heating during the time the temperatures of the substrate and the crucible are being stabilised, but the moment the shutter is moved to expose the substrate to the effusion aperture, the substrate gets exposed to direct radiation from the tungsten coil. This effect is minimised if the distance between the crucible and the substrate is large. In a bell jar of 12" diameter, this distance could be from 6 to 8".

Using conventional boats, it is difficult to obtain good quality films of metals, like *Sb* under normal conditions of vacuum, especially when the substrate is at an elevated temperature. It has been demonstrated[8,9] that with the help of this crucible, good films of *Sb* can be obtained quite conveniently.

In this type of the Knudsen source, the lid is press-fitted on the crucible. When heating takes place the vapour pressure inside the crucible is higher than the outside pressure. The vapours keep on escaping from the effusion aperture of the crucible and condense on the shutter.

The films prepared by using this evaporation source give consistent results on various materials mentioned earlier. Figure 2 illustrates *CdSe* film prepared by vacuum evaporation from this source under captioned conditions of evaporation. The electron micrograph was taken on Siemens Elmiskop 1 operated at 80 KV. Photoconductive response of a 1500 Å thick *CdSe* film obtained by vacuum evaporation from this source has been measured. The results of these photoconductive measurements will be published soon.

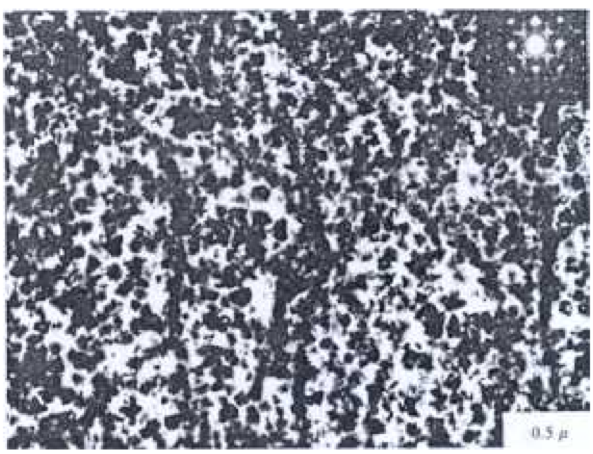

Figure 2. Electron micrograph of a continuous *CdSe* film : 350 Å thick deposited onto *KCl* at 300 °C: rate of deposition 50 Å/s. The corresponding SAD is shown in the inset.

An important advantage of this source is that the substrate and the substrate heater are situated at the base of the chamber and the evaporated material travels from the aperture of the crucible in the downward direction as compared to other conventional sources, where the material evaporates upwards onto the substrate, which is kept inverted. Hence, the problems associated with the inverted substrate are avoided. Yet another advantage of this crucible is its long life because the problems of wetting and alloying, which exist normally with metallic boats are avoided in most of the evaporants in this crucible. However, it is advisable to use a separate crucible for each evaporant to avoid any cross contamination.

The rate of evaporation of the material can be increased by increasing the temperature of the crucible, which in turn can be increased by increasing the current through the tungsten coils. With use, these coils become brittle and ultimately give way. In that case one has only to change the heating coil and the intact crucible is ready for the next run.

4. CONCLUSION

This modified Knudsen type crucible is simple, long lasting and can be used for the routine vacuum evaporation of almost all types of sublimating materials up to 1000 °C.

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