

## Preparation and Properties of Niobium Thin Films

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**ABSTRACT.** Niobium thin films have been prepared by thermal evaporation techniques in a UHV system using an electrostatically focussed electron beam evaporator. The starting material was MARZ grade niobium, with 99.999% purity, and in discs 12 mm diameter by 3 mm. Deposition was, in general, on to thermally oxidised silicon slices with deposition rates of the order of  $2 \text{ \AA s}^{-1}$ .

Film properties were examined as a function of film thickness, deposition rate and substrate temperature during deposition. The critical temperature ( $T_c$ ) was assessed by four-probe assessment of the sheet resistivity ratio at room temperature and in liquid nitrogen. These indicated a critical temperature close to the bulk value of 9.2 K appropriate to niobium and, in all cases, the films were superconducting in liquid helium (4.2 K).

Films of thicknesses  $< 500 \text{ \AA}$  revealed  $T_c$  values  $< 9.2 \text{ K}$  and the film resistivity was significantly greater than should have been expected. This was thought to be due to incomplete coalescence of the nucleation processes and a contribution to surface scattering in electron transport.

### Introduction

Niobium is not an easy material from which good quality films can be prepared. In particular, it is a refractory metal with a very high melting point and low vapour pressure requiring an evaporation source temperature of more than 300 K. Much effort has been invested in order to produce clean niobium films with  $T_c \geq 9 \text{ K}$ . It is well

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known<sup>[1]</sup> that oxygen is the main source for depression of the superconducting properties of niobium and techniques must be chosen to restrict the oxygen concentration,  $< 0.1\%$ , since the depression of  $T_c$  will then be  $< 0.1$  K.

It is well known that, if unit sticking probability is assumed, a monolayer of adsorbate will be formed approximately per second<sup>[2]</sup> if the partial pressure of the adsorbing species is  $10^{-6}$  mbar. Since the film growth rate of the niobium is only expected to be of the order of a monolayer every 10 seconds it is clear that the oxygen partial pressure must be less than  $10^{-9}$  mbar. Such a pressure requires the most stringent vacuum environment which is the reason that UHV techniques were chosen.

### Experimental

Niobium is a metal that getters gaseous species when it is freshly deposited on to a surface with the result that its superconducting properties may be adversely affected and render it useless for any superconducting application<sup>[3]</sup>. This imposes the need for a clean deposition environment so that a UHV system has been purposely designed for the task.

Figure 1 shows, schematically, the arrangement of the UHV system that was developed. It is essentially comprised of two chambers interconnected by a gate valve so that they can be individually pumped to prevent cross contamination. One of the

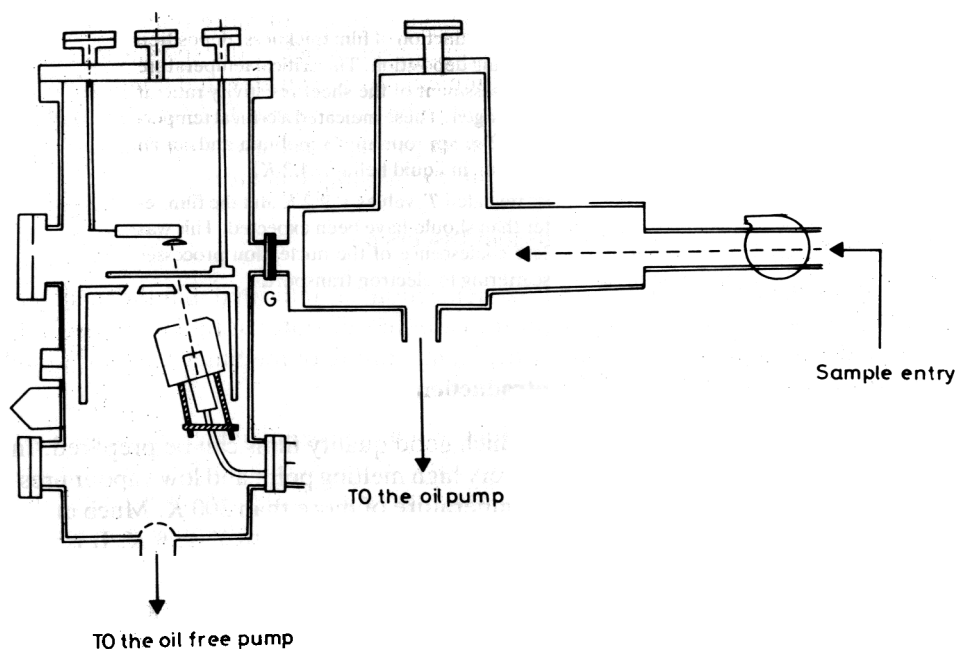


FIG. 1. Schematic diagram for UHV system.

chambers is continuously maintained at UHV by pumping with a combination of a  $250 \text{ ls}^{-1}$  diode ion pump which is supported, during metal layer deposition, with a liquid nitrogen baffled titanium sublimation pump. The sublimation pump has an effective speed of  $1000 \text{ ls}^{-1}$  which makes it ideal to keep the pressure low during any high gas load phase, such as evaporation.

The evaporation sources are maintained above the baffle for the sublimation pump and below a further liquid nitrogen cooled baffle which has holes to allow the evaporant to reach the substrate. In this way the substrate is always maintained in a clean environment with minimal chance of contamination.

Substrates are entered into the system using a diffusion pumped preparation chamber. This pump has a combination liquid nitrogen trap and bakeable valve which allows rapid, clean pump down from atmosphere and the facility for handling high gas loads. It is also capable of coping with inert gases which makes it complementary to the ion pump used in the deposition chamber.

Bakeout of both chambers is achieved using internally mounted quartz halogen lamps which offer a rapid and convenient way of cleaning the system. Figure 2 shows a typical mass spectrum of the residual gases, taken with a VSW Scientific Instruments Ltd. Micromass quadrupole mass spectrometer, within the preparation chamber where the background requirements are less stringent. It indicates that the system is essentially free from contaminants except hydrogen, water and some hydrocarbons.

Except for hydrogen, no species has a partial pressure of more than  $10^{-12} \text{ mbar}$ , and there is no indication of the presence of oxygen which is the species most concern. The spectrum was taken with the sample heated to an elevated temperature of  $310^\circ\text{C}$  when the gas load would be expected to be at its worst. Indeed, this explains the presence of hydrocarbons which are most probably release from the coating of the leads used to carry the electrical current to the sample heater.

## **Results and Discussion**

The effect of substrate heating during deposition has indicated that the quality of the film is substantially improved over the cases of substrates at room temperature. An established quality factor is the critical temperature of the film which was found to be  $9.2 \text{ K}$ .

Measurement of this for a number of films is tedious, so advantage has been taken of the finding that the film sheet resistance ratio of measurement at room temperature and  $77 \text{ K}$  can be correlated with critical temperature<sup>[4]</sup>. It has been reported that a ratio in excess of 2 can be correlated to a critical temperature of pure niobium.

Experiments were carried out for two different substrate temperatures,  $50^\circ\text{C}$  and  $300^\circ\text{C}$ , with total pressure of the order of  $10^{-9} \text{ mbar}$ . The thickness of the deposited film is monitored with a quartz crystal, water cooled, placed inside the vacuum chamber. Films of about 500, 1000 and 2000  $\text{Å}$  were deposited on  $\text{SiO}_2$  substrates.

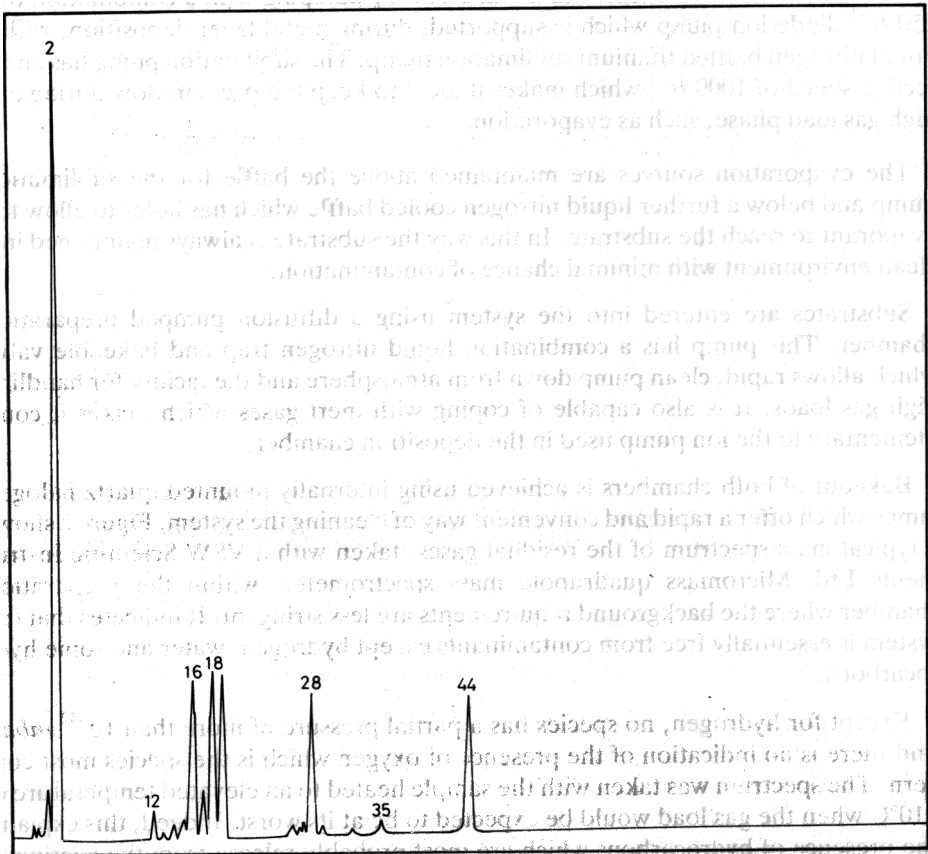
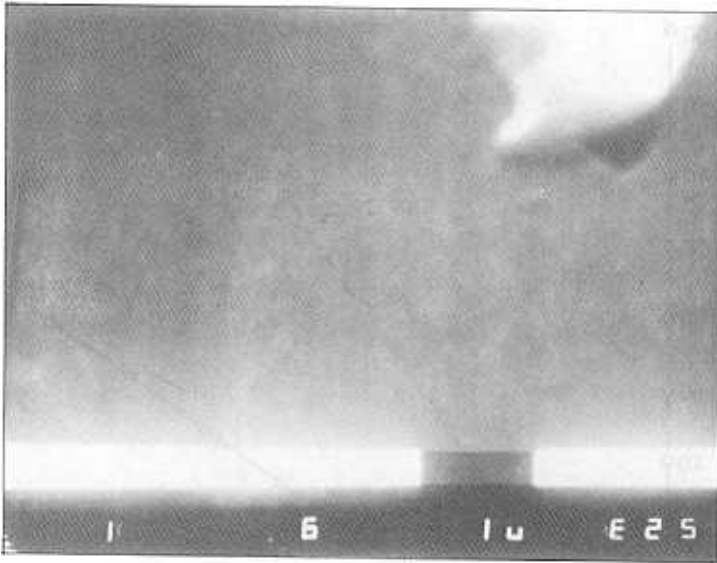


Fig. 2. Typical residual gas spectrum with V.S.W. quadrupole mass analyser. Total pressure  $5 \times 10^{-10}$  m.bar, S.E.M.  $10^{-13}$  m.bar.

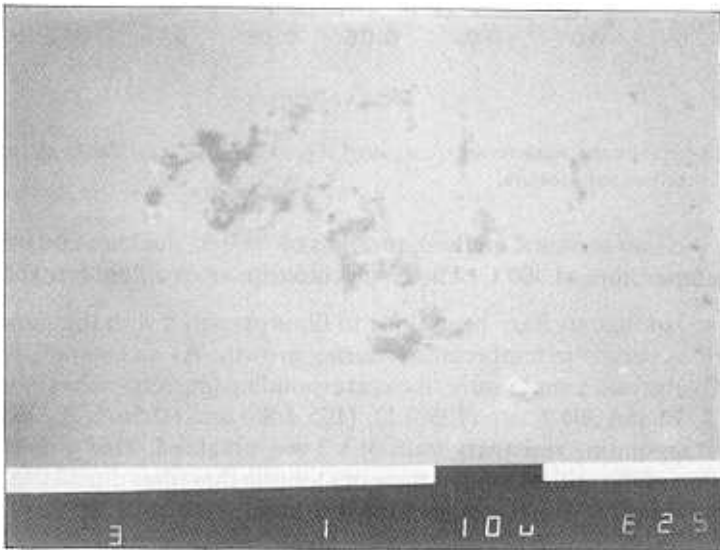
Figures 3 and 4 show SEM micrographs of two films with thicknesses of 500 and 2000 Å, respectively. As indicated in Figure 4 the film shows some residual structure from the substrate indicating that the grain size is of the order of the film thickness. There are also indications of discontinuity in the film thickness and that the films are not very smooth.

X-ray examination shows that both films have a polycrystalline structure. It is observed that the thinner films are more heavily influenced by the substrate surface but, as film thickness increases ( $> 2000$  Å) continuous films (with no substrate influence) were obtained.

Electrical measurements of the thinnest films showed very poor resistance ratios of less than unity even when the film was prepared at very high substrate temperatures. Figure 5 shows the current-voltage characteristics, measured at various tempera-



**FIG. 3.** S.E.M. micrograph of NB films with thicknesses of 2000 Å.



**FIG. 4.** S.E.M. micrograph of NB films with thickness of 500 Å.

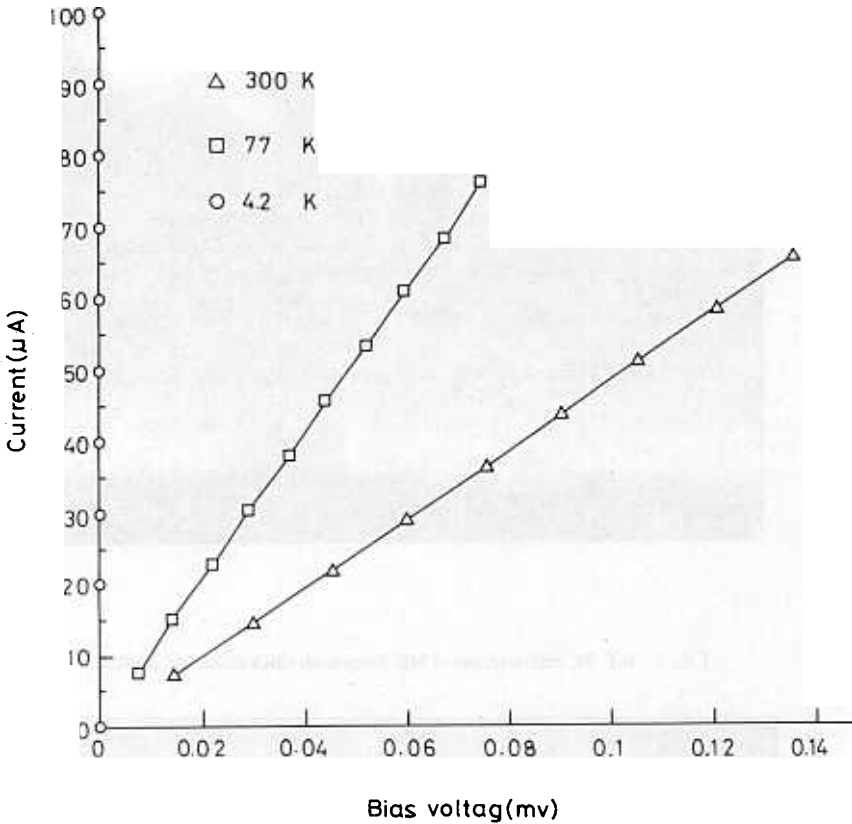


FIG. 5. The current-voltage characteristics, measured at (○) 4.2, (◇) 77, (△) 300°K, of the Nb film with 300°C substrate temperature.

tures using the four terminal method, for films of 2000 Å thickness and prepared at a substrate temperature of 300°C. The results indicate an excellent ratio of 2.25<sup>[5]</sup>.

Similar measurements have been done to films prepared with the same thickness but for various substrate temperatures during growth. As an example for niobium film of 50°C substrate temperature, the corresponding (current-voltage) values measured at 4.2, 77 and 300°K are (105,0.1), (105,1.85) and (105μA, 2.4 mV), respectively. A disappointing resistivity ratio of 1.3 was obtained. This is consistent with the depression of the critical temperature of niobium thin films due to use of low substrate temperature<sup>[6,7]</sup>. Thus the higher substrate temperature of 300°C is favourable.

### Conclusion

In order to grow high quality niobium films, at least three parameters must be considered which are substrate temperature, residual gas pressure and film thickness. It

has been found that these parameters are crucial in determining the film quality. The present study has shown that the best quality films have been obtained at a substrate temperature of 300°C and a thickness of 2000Å.

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## إعداد وخصائص أغشية رقيقة من مادة النيوبيوم

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المستخلص . أُعدت أغشية النيوبيوم الرقيقة (Nb) في صورة أقراص بأقطار ٣ مم ، ١٢ مم بواسطة التبخير الحراري في وسط فائق التفريغ وذلك باستخدام قاذفة إلكترونيات . الغرض من هذا التبخير هو الحصول على أغشية رقيقة ذات درجة نقاء من الشوائب عالية جداً ولا تقل عن ٩٩,٩٩٩٪ وقد تم فحص هذه الأغشية كهربائياً في ظروف مختلفة وفي درجات حرارة متفاوتة ٤٢ ، ٧٧ ، ٣٠٠ درجة كلفن ووجد أن الأغشية الرقيقة التي يقل سمكها عن ٥٠٠ أنجستروم غير مناسبة للتطبيقات العملية وذلك لردائتها توصيلها الكهربائي وعدم ثباتها في حالة مستقرة .

أما الأغشية التي يزيد سمكها عن ذلك فقد وجد أنها تظل ثابتة الخواص كهربائياً وفيزيائياً ولا تتأثر بالعيوب المحتمل وجودها في سطح القاعدة .

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