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REFLECTANCE OF GALLIUM FILMS IN THE WAVELENGTH RANGE FROM 0.25 μm TO 25 μm

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Experiments were made with gallium films condensed on cooled glass and quartz substrates under a vacuum of $p = 1\times10^{-5}$ torr. The reflectance coefficient of the gallium films was determined for normally incident light within the wavelength range from 0.25 μm to 25 μm. The deposition rate of the gallium films was 50 Å/sec. It was found that a dendrite structure appears in thin films condensed on cooled substrates.

1. Introduction

In earlier work [1, 2, 3] research was done on the optical properties of thin gallium films evaporated under high vacuum and condensing on a substrate at room temperature. It was found then that slow deposition of gallium films alone enables the achievement of films with metallic lustre. It was also noticed that under such conditions it is impossible to obtain very thick opaque gallium films with metallic lustre. Owing to the low melting point of gallium, 29.78 °C, it seems reasonable to evaporate gallium films in vacuo on a cooled substrate. Therefore, studies on gallium films condensing on a cooled substrate and their optical properties were continued.

2. The process of evaporating gallium films on a cooled substrate

The gallium films were evaporated in a vacuum apparatus at a pressure of $p = 1\times10^{-5}$ torr.

Fig. 1, shows a diagram of the device cooling the substrate, mounted inside the vacuum apparatus, onto which the gallium film is evaporated. The cooling device has the form of a rounded cylinder A of height 6 cm and diameter 20 cm to which there are rigidly attached three rods E leaning against the windows of the bell B. A Monel metal tube C, the free end

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of which is led out into the atmosphere through the window of the apparatus bell, is connected to the cylinder. This Monel metal tube is used for pouring liquid air or liquid oxygen into the cylinder. The copper plate (*D*) with windows in which the substrates for the gallium films are located adheres to the bottom of the cylinder, clamped down by screws. In order to obtain the required lowering of temperature use was made of rubber washers or a special vacuum grease between the substrate and cylinder. The temperature of the base was measured by means of a copper-constantan thermocouple, one joint of which was glued with BF2 adhesive to the substrate, while the other was maintained at a temperature of 0 °C. The terminals of the thermocouple were connected to a KTP and compensator. Herasil fused quartz (manuf. Heraeus) and BK7 glass were used as the substrates for the films. The sub-

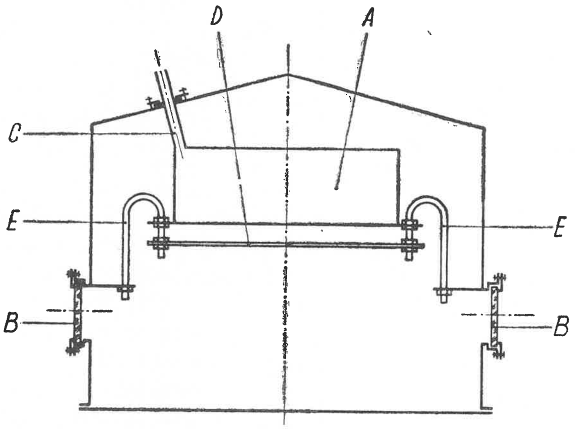

Fig. 1. Diagram of device cooling substrate. *A* — rounded cylinder, *B* — bell window, *C* — Monel metal tube, *D* — copper plate, *E* — rods supporting container

strates had the form of optical wedges of an angle of 5° and/or plane parallelplate s of the dimensions 25 mm × 35 mm. The heaters evaporating the gallium were a tungsten basket aoated with alundum Al_2O_3 in an electrophoretic process and sintered in a hydrogen furnace ct 1400 °C, a tungsten boat and a graphite boat. The graphite heater proved to be inappropriate owing to this pulverization of the graphite. As in the earlier work the gallium was of Chinese manufacture and had a purity of 99.9886 per cent. The rate of depositing the gallium was increased considerably as compared with the earlier work [1, 2, 3]. This rate now equalled 50 Å/sec, instead of the previous several angstroms per minute.

3. *Measurement of reflectance coefficients and film thicknesses*

The measurements of the reflectance coefficient *R* and the gallium film thickness from the gallium-air side were conducted for normal incidence. The measurements within the different spectral ranges were conducted with different instruments. In the 0.25 μm to 1 μm wavelength range the measurements were made with a Zeiss VSU-1 spectrophotometer with a reflectance unit [1]. In the 1 μm to 2 μm range a 203 microvoltmeter with a PbS photoresistor coupled with a VSU-1 monochromator was used. The 2 μm to 25 μm range was covered by means of a UR-20 spectrophotometer. Details of the measurements are specified in Table I.

In the measurements of the reflectance coefficient with the UR-20 spectrophotometer use was made of two reflectance units designed and built by R. Brunsch, M. Sci. The path

TABLE I

Wavelength range	Light source	Monochromator	Detector	Substrate
0.25 μm to 0.4 μm	hydrogen lamp	mirror type, Zeiss, NACl prism	MQVS vacuum photocell	Herasil fused quartz
0.4 μm to 0.6 μm	tungsten filament lamp	mirror type, Zeiss, glas prism	MQVS vaccum photocell	silicate rod, glass or fused quartz
0.6 μm to 1 μm	tungsten filament lamp	mirror type, Zeiss, glas prism	MV photocell PbS	
1 μm to 2 μm	radiation, modulation	mirror type, Zeiss, glas prism	photoresistor	
2 μm to 25 μm	silicate rod, radiation, modulation	UR-20 monochromator		

of the rays in the reflectance unit is shown diagrammatically in Fig. 2. The beam of light leaving the source, after reflection from the aluminum mirror Z_1, falls under an angle $\alpha < 10°$ on the examined film W or an aluminum reference film. Next, it is again reflected at the second mirror Z_2 and then follows the ordinary path in the UR-20 spectrophotometer.

The reflectance units were placed in the path of two radiation beams: one in the reference beam (J_0), the other in the measurement beam. Two reflectance units were used in order to ensure an identical optical path of the two beams of radiation in the spectrophotometer.

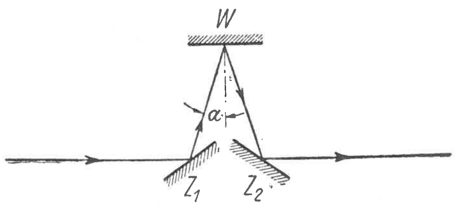

Fig. 2. Diagram of ray path in reflectance unit. Z_1, Z_2 — aluminum mirrors, W — examined film or aluminum mirror

The measurements of the reflectance coefficient, in the entire spectral range, were relative measurements with respect to silver or aluminum reference films. These reference films were evaporated under a vacuum of $p \approx 1 \times 10^{-6}$ torr. The quality of the references wa; checked by measuring the absolute reflectance coefficient R in the visible spectral ransge the results were in conformity with data in the literature [4]. The accuracy of reflectance coefficient R measurements was ± 1 per cent.

The thickness of the gallium films was determined by the multiple beam interference technique by means of an interference microscope [5].

4. *Results of measurements and discussion*

Figs 3 and 4 present the dependence of reflectance coefficient R on wavelength λ for the opaque gallium film evaporated from the tungsten boat. Fig. 3 depicts the function $R = f(\lambda)$ for the range from 0.25 μm to 5 μm, Fig. 4 in the 2 μm to 25 μm range. The 0.27 μm thick film of gallium was deposited on a quartz substrate cooled to a temperature of -100 °C.

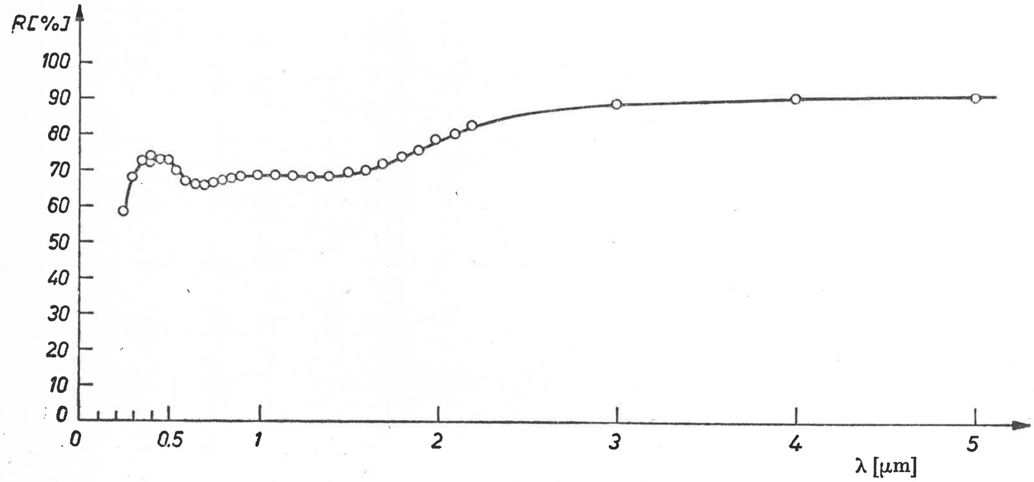

Fig. 3. Reflectance coefficient R of 0.27-μm thick gallium film *versus* wavelength

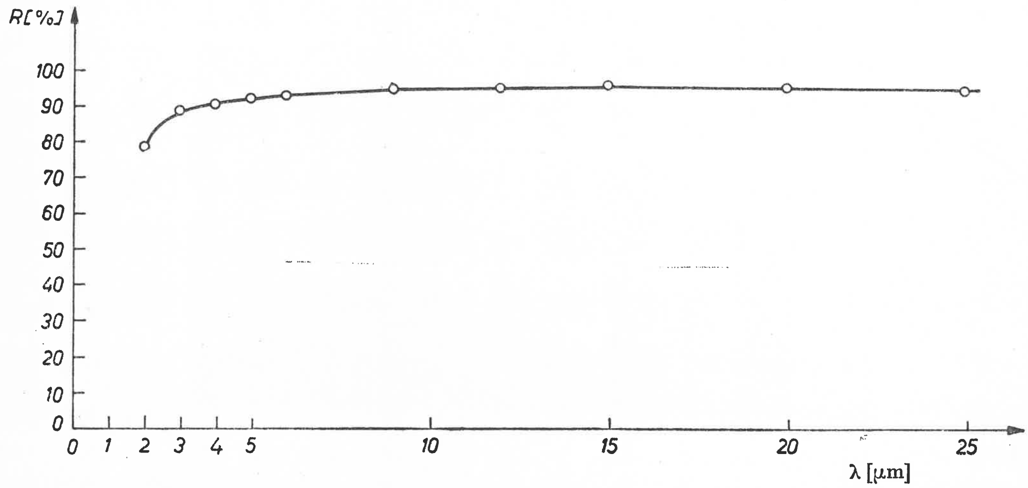

Fig. 4. Reflectance coefficient R of 0.27-μm thick gallium film *versus* wavelength

It displayed a metallic lustre over its entire area (25 mm × 35 mm), without any cracks or dendrites. The reflectance coefficient for gallium is seen to increase distinctly from these figures in the far infrared region, what is a characteristic feature of some other metals, also. In the visible and near infrared regions there are extremes (a maximum and minimum) of reflectance coefficient. An absorption band, which appears in the case of a gallium single crystal [6, 7] and a polycrystalline gallium film [8, 9], is to be expected in this region.

The characteristic features of the $R = f(\lambda)$ curves described above appeared for many gallium films condensing on cooled substrates, independently of the evaporation rate. On the other hand, the reflectance coefficient of gallium films in the near ultraviolet region depends on the evaporation process.

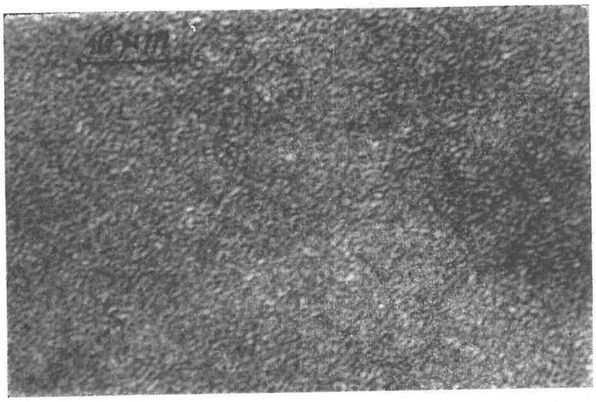

Fig. 5. Grains of gallium film (magnification 1500×)

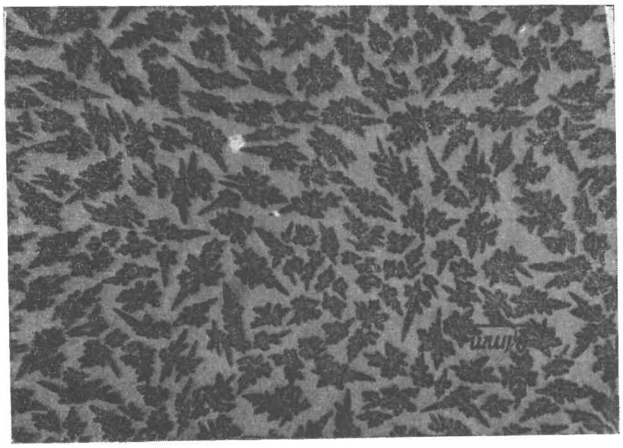

Fig. 6. Dendrites of gallium film (magnification 135×)

The performed investigations on films deposited on cooled substrates make it possible to ascertain that opaque gallium films with metallic reflection may be obtained in a fast evaporation process only on a substrate cooled to a temperature below −50 °C. If the temperature is not low enough the thick films of gallium acquired in a fast evaporation process are milky and dull, while metallographic microscopy show them to have a granular structure like that shown in Fig. 5. On the other hand, thin gallium films display a dendrite structure like that shown in Figs 6 and 7. It was found that the formation of gallium dendrites is independent of the type of heater used (tungsten boat, graphite boat or Al_2O_3 coated tungsten basket). It was also noticed that thick gallium films condensing on a substrate at a temperature near that of liquid O_2 become cracked after transition to room temperature.

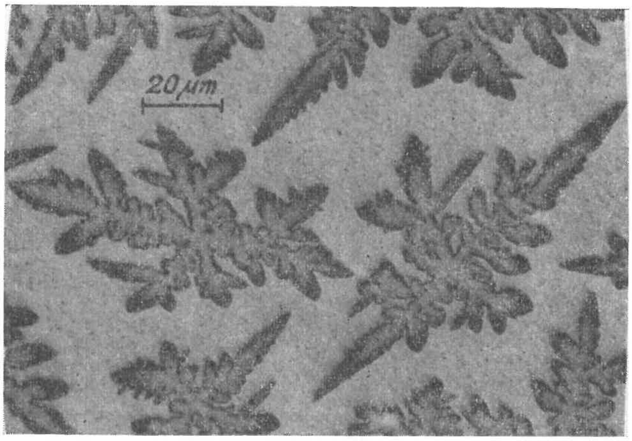

Fig. 7. Dendrites of gallium film (magnification 540×)

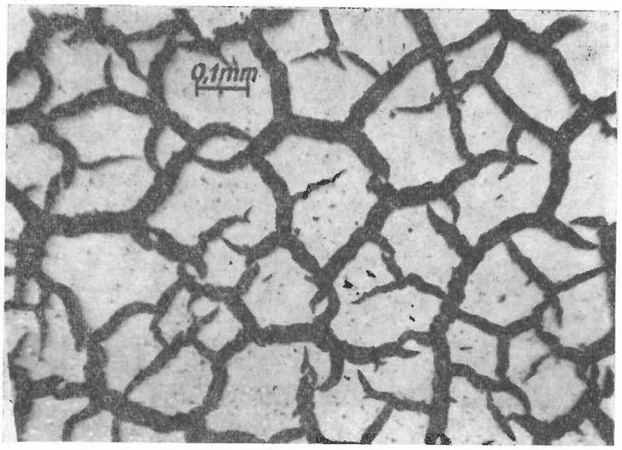

Fig. 8. Cracks in gallium film (magnification 70×)

Further research on gallium films condensing on cooled substrates is in progress.

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