

# Apparatus for the controlled deposition of multilayer films

Tsukasa Sawaki, Minoru Iwata, Shizuko Katsube, Kazuo Hara

► **To cite this version:**

Tsukasa Sawaki, Minoru Iwata, Shizuko Katsube, Kazuo Hara. Apparatus for the controlled deposition of multilayer films. *Journal de Physique*, 1964, 25 (1-2), pp.258-261. <10.1051/jphys:01964002501-2025800>. <jpa-00205752>

**HAL Id: jpa-00205752**

**<https://hal.archives-ouvertes.fr/jpa-00205752>**

Submitted on 1 Jan 1964

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

## APPARATUS FOR THE CONTROLLED DEPOSITION OF MULTILAYER FILMS

By TSUKASA SAWAKI,

Research Laboratory, Matsushita Electronics Corporation, Takatsuki, Osaka, Japan.

MINORU IWATA and SHIZUKO KATSUBE,

Government Industrial Research Institute, Ikeda, Osaka, Japan.

KAZUO HARA,

Vacuum Optics Corporation of Japan, Higashi-togoshi 1, Sinagawa, Tokyo, Japan.

**Résumé.** — On décrit un appareillage simple et stable pour contrôler la préparation de couches multiples sous vide. De façon à comparer les facteurs de transmission ou de réflexion d'une couche multiple pour deux longueurs d'onde, on utilise un faisceau lumineux modulé, deux filtres interférentiels, deux photomultiplicateurs avec amplificateurs et un amplificateur différentiel. On a utilisé l'appareil dans un domaine de longueur d'onde s'étendant de l'ultraviolet au proche infrarouge, et on a adapté de manière convenable la source lumineuse et le photomultiplicateur. La reproductibilité des dépôts était de  $\pm \lambda_0/65$  pour le filtre à bande étroite d'un film à 13 couches.

**Abstract.** — A simple and stable apparatus for the controlled deposition of multilayer films in vacuo is described. In order to compare transmittance or reflectance of a multilayer film at two wavelength bands, the apparatus is equipped with a modulated light beam, two interference filters, two photo-tubes with amplifiers and a differential amplifier. The apparatus has been used in a wavelength range from the ultraviolet to the near infrared with suitable adaptation of the light source and the photo-tubes. Reproducibility of the deposition was  $\pm \lambda_0/65$  for the narrow band-pass filter of a thirteen-layer film.

**1. Introduction.** — In the field of applied optics a great deal of interest has recently been shown in the properties of multilayer interference films for use as high-reflecting mirrors, band pass filters, etc. To meet this requirement, precise control of film thickness in multi-layer films is very important. For the controlled deposition of multilayer films the photometric method seems to be most convenient, though various methods for the controlled deposition of thin films in vacuo have been described by many authors [1] a [6]. An apparatus for the controlled deposition of multilayer films constructed by the present authors is based on the comparison of transmittances or reflectances of a multi-layer film for two beams of different wavelength during the deposition [7]. The apparatus can be used in the near infrared after a simple replacement of photo-tubes by photoconductive cells, and can also be used in the ultraviolet by applying optical materials transparent to ultraviolet light.

**2. Design considerations and construction of the apparatus.** — The transmittance or reflectance curve of a stack of quarter-wave films, alternately of high and low index, plotted as a function of wave number is symmetrical with respect to the central wave number [1]. When the optical thickness of the top layer of the multi-layer films is increasing from zero to a quarter wavelength, trans-

mittance or reflectance of the resultant film varies with the thickness in a regular way as will be shown in a later chapter. Therefore, the apparatus was designed in such a way it could measure the difference of transmittances or reflectances of the resultant film at two wavelengths, each of which is situated at a suitable and equal wave number distance from the central wavelength. During the deposition, the thickness of each individual layer can be controlled by observing the indicating meter, which shows subsequent zero differences at those moments when the optical thick-

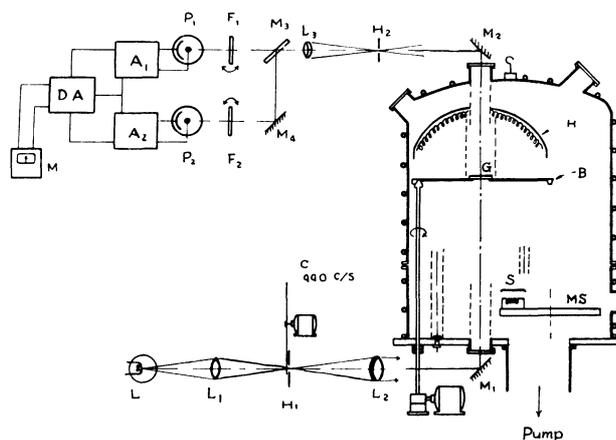


FIG. 1.



of quarter-wave films, alternately of high and low index, is controlled by a transmitted light beam of normal incidence [7]. Writing  $\delta_k$  for the change in phase of light of wavelength  $\lambda$  on traversing the  $k$ -th film of optical thickness  $(nt)_k$ , we have

$$\delta_k = 2\pi(nt)_k/\lambda. \tag{1}$$

With the apparatus the transmittances ( $T_k$ ) in two wavelength bands are compared with each other. They become equal when the  $k$ -th layer just becomes a quarter of the central wavelength  $\lambda_0$ . Writing  $\lambda_1$  and  $\lambda_2$ , for the central wavelengths of two observation bands, we obtain for the sensitivity  $S_T$  of the deposition control the following expression :

$$S_T = \left| T_k \times \frac{\partial T_k}{\partial (nt)_k} \right|_{(\lambda=\lambda_1)} + \left| T_k \times \frac{\partial T_k}{\partial (nt)_k} \right|_{(\lambda=\lambda_2)}$$

$$= \frac{2\pi}{\lambda_0} \times T_k \times \left| \frac{\partial T_k}{\partial \delta_k} \right| \times \left\{ \frac{\lambda_0}{\lambda_1} + \frac{\lambda_0}{\lambda_2} \right\} \tag{2}$$

$$= \frac{4\pi}{\lambda_0} \times T_k \times \left| \frac{\partial T_k}{\partial \delta_k} \right|. \tag{3}$$

On each side of the so-called pass-band of the resultant  $k$ -layer film, there are a principal maximum and ( $k - 1$ ) secondary maxima of  $\partial T_k/\partial \delta_k$ . However, variation of transmittance around secondary maxima of  $\partial T_k/\partial \delta_k$  is so complicated that it can not be used for the deposition control, as is apparent from (fig. 3), which shows the change of

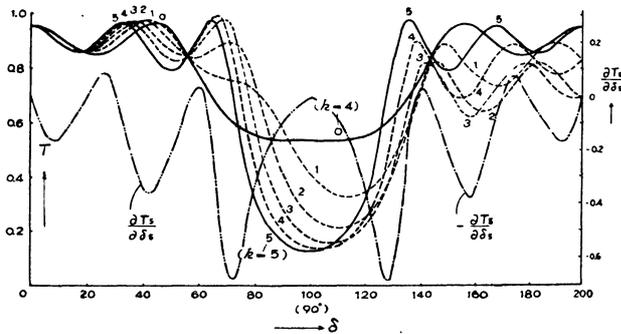


FIG. 3.

the calculated transmittance with a stepped increases of  $\lambda_0/20$  of the top-layer's thickness of five-layer film of quarter-wave films, alternately of ZnS ( $n = 2.35$ ) and  $MgF_2$  ( $n = 1.40$ ). In figure 4, spectral transmittance of the  $k$ -layer film and spectral sensitivity of the deposition control are shown. Although different filters for each individual layer are theoretically required, it is more practical to choose the observation filter for the last layer and use this throughout the deposition of the subsequent layers, since exchange of filters to the most fitting ones for these layers during the deposition is very difficult.

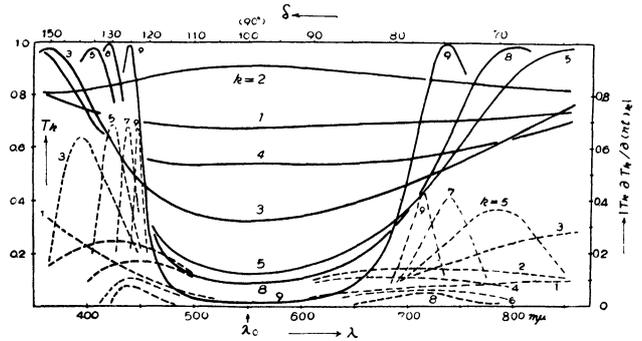


FIG. 4.

**4. Application of the apparatus.** — It was found that the deposition of a stack of quarter-wave and three-quarter-wave films, alternately of high and low index, could be controlled with the apparatus in a similar way as the deposition of a stack of quarter-wave films. In order to control the deposition of a narrow band-pass filter containing a spacer layer of a half-wave film with the apparatus, a monitoring substrate had to be exchanged to a new one before and after, or in the middle of the process of depositing the spacer layer. This seems to be based on the fact that after deposition of the spacer layer a narrow pass-band appears around the central wavelength and useful regularity of variation of transmittance for the deposition control appears only around the narrow pass-band. Reproducibility was within  $\pm \lambda_0/65$  for the deposition of the first-order narrow band-pass filter of a thirteen-layer film of ZnS and  $MgF_2$  [7].

The deposition of heat reflecting films [9] could be controlled satisfactorily by means of replacing one of the photo-tube with a PbS photo-conductive

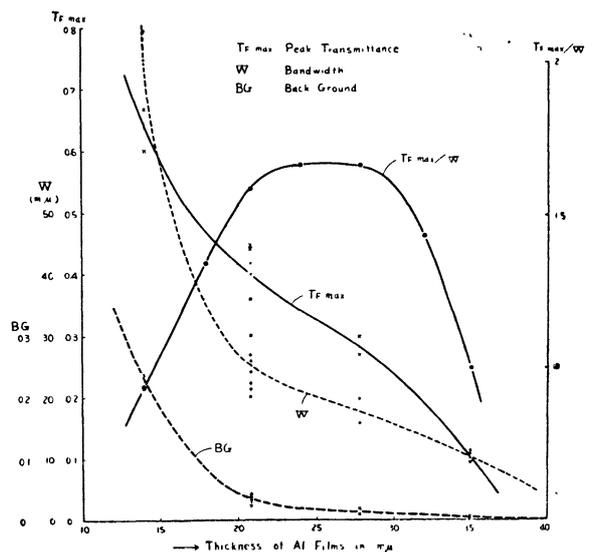


FIG. 5.

cell which is more suitable for the longer wavelengths. The deposition of high-reflecting films for the near infrared, such as films for gas lasers [10], could also be controlled with exchanging two phototubes for two photo-conductive cells. For these purposes the authors employed  $\text{CeO}_2$  for high index films and  $\text{MgF}_2$  for low index films.

The apparatus has also been applied for the deposition of multi-layer films for the ultraviolet by means of replacing the white light lamp with a hydrogen discharge tube, and optical glass with

fused quartz. Interference filters for observation bands in the ultraviolet were made of Al films and  $\text{MgF}_2$  films. Measured characteristics around 300  $\text{m}\mu$  of the second-order filters are shown in figure 5 as a function of the thickness of Al films. On the basis of this preliminary experiment, thicknesses of Al films were chosen to be about 25  $\text{m}\mu$ .

High reflecting films for the Fabry-Perot interferometer in a wavelength range from 300  $\text{m}\mu$  to 450  $\text{m}\mu$  were made with  $\text{Sb}_2\text{O}_3$  films and  $\text{MgF}_2$  films. A non-absorbing film of  $\text{Sb}_2\text{O}_3$  is deposited with a metal boat source covered with alumina by means of flame-spraying [11]. Some of the results are shown in figure 6.

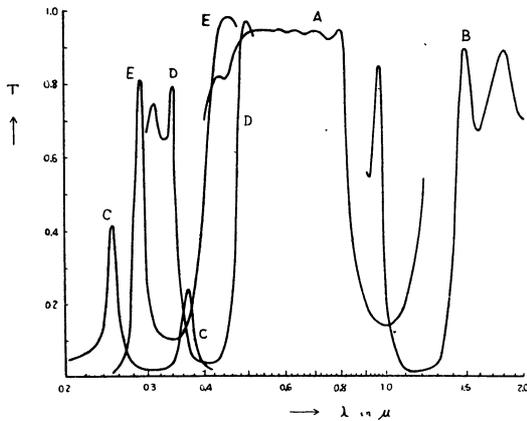


FIG. 6. — A: 8-layer film of  $\text{CeO}_2$  ( $\lambda/8$ ) —  $\text{CeO}_2$  ( $\lambda/4$ ) —  $\text{MgF}_2$  ( $\lambda/4$ ) . . . . . for heat reflecting filter, B: 11-layer film of  $\text{CeO}_2$  ( $\lambda/4$ ) —  $\text{MgF}_2$  ( $\lambda/4$ ) . . . . . for high-reflecting film for gas laser. C: Interference filter of Al —  $\text{MgF}_2$  — Al. D: 11-layer film of  $\text{Sb}_2\text{O}_3$  ( $\lambda/4$ ) —  $\text{MgF}_2$  ( $\lambda/4$ ) . . . . . for Etalon. E: 7-layer film of  $\text{Sb}_2\text{O}_3$  ( $\lambda/4$ )  $\text{MgF}_2$  ( $\lambda/4$ ) . . . . . for a narrow band filter.

5. **Conclusions.** — Design and construction of apparatus for the controlled deposition of multi-layer films are described together with the results. Adoption of the photometer system with two photo-tubes enabled the apparatus to be used in a wavelength range from the ultraviolet to the near infrared. Adoption of interference filters for observation bands and of the method of gain-control of the amplifiers for the zero adjustment of the deposition also made the apparatus simple and stable.

However, for a narrow band-pass filter the reproducibility of  $\pm \lambda_0/65$  is not so satisfactory as that of more complicated apparatuses [4]. This can however be considerably improved with improvements of stabilized voltage supply for the light source and of the signal-to-noise ratio of the photometer amplifier. A further study based on this consideration is now in progress.

#### BIBLIOGRAPHY

- [1] HEAVENS (O. S.), "Optical Properties of Thin Solid Films", Academic Press, 1955.  
HOLLAND (L.), "Vacuum Deposition of Thin Films", John Wiley, 1956.
- [2] LISSBERGER (P. H.) and RING (J.), *Optica Acta*, 1955, **2**, 42.  
GIACOMO (P.), *Rev. d'Opt.*, 1956, **35**, 317.
- [3] TRAUB (A. C.), *J. Opt. Soc. Am.*, 1956, **46**, 999.  
MORI (R.) and TAKAOKA (T.), *J. Appl. Phys.*, Japan, 1957, **24**, 443.
- [4] STECKELMACHER (W.), PARISOT (J. M.), HOLLAND (L.) and PUNTER (T.), *Vacuum*, 1951, **9**, 171.  
STECKELMACHER (W.) and ENGLISH (J.), 1961, Vacuum Symposium Transaction, 852, Pergamon, 1962.
- [5] LOSTIS (P.), *Rev. d'Opt.*, 1959, **38**, 1.  
SAUERBREY (G.), *Z. Physik*, 1959, **155**, 206.
- [6] GIEDD (G. R.) and PERKINS (M. H.), *Rev. Sc. Instr.*, 1960, **31**, 773.  
SCHWARZ (H.), *Rev. Sc. Instr.*, 1961, **32**, 194.
- [7] SAWAKI (T.), IWATA (M.) and KATSUBE (S.), *J. Vac. Soc.*, Japan, 1960, **3**, 304.
- [8] VALLEY (G. E.) and WALLMAN (H.), "Vacuum Tube Amplifiers", McGraw-Hill, 1948, p. 384.
- [9] SCHRÖDER (H. H.) and TURNER (A. F.), *J. SMPTE*, 1960, **69**, 351.
- [10] JAVAN (A.), BENNETT (W. R.) and HERRIOTT (D. R.), *Phys. Rev. Letters*, 1961, **6**, 106.
- [11] SAWAKI (T.), *Science of Light*, Japan, 1958, **7**, 1.