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Study on defects in ZnS/YbF_3 infrared coatings on silicon substrates

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ABSTRACT

In order to reduce the numbers of defects, the influence of deposition parameters (i.e., deposition rate and substrate temperature) and deposition methods (i.e., thermal evaporation and electron beam evaporation) on the defect density of ZnS monolayer, YbF_3 monolayer and ZnS/YbF_3 multilayer coatings deposited on silicon substrates were investigated experimentally. The results show that deposition rate and substrate temperature have major influence on the defect density. For ZnS monolayer coatings, the optimum deposition rate is approximately 1.2 nm/s, and the optimum substrate temperature is from 80 °C to 120 °C. Meanwhile, for YbF_3 monolayer coatings, the optimum deposition rate is approximately 0.4 nm/s, and the optimum substrate temperature is from 100 °C to 150 °C. Furthermore, the defect density of ZnS or YbF_3 monolayer coatings deposited by thermal evaporation is much lower than that by electron beam evaporation. The defect density of ZnS/YbF_3 multilayer coatings can be reduced significantly by optimizing the substrate temperature and deposition rate with the method of thermal evaporation.

1. Introduction

Defects in infrared coatings are one of the most important factors influencing the optical performance and stability of infrared coatings, and are always major concerns [1]. Defects in multilayer coatings have been investigated extensively for many years [2–6]. These defects are created by particulates, either on the substrate surface before coating or deposited during the coating process [7]. It is believed that the defects, such as contaminants, impurities, and void fillers whose size could range from a few to several hundred nanometers, are included in the coatings due to the impurity of the raw material and inhomogeneous film growth during the deposition process [8,9]. Clean-room conditions are found to be essential for cleaning and coating low-defect substrates. In general, the levels of surface and subsurface defects on the substrate are the most important parameters in determining the defect density in a prepared coating [10].

In this paper, ZnS single layer, YbF_3 single layer and ZnS/YbF_3 multilayer coatings deposited on silicon substrates were prepared under different deposition parameters and methods. The defects of these coatings were counted and the defect density was determined. By investigating the dependence of the defect density on the deposition parameters and deposition methods, the optimum substrate temperature and deposition rate, as well as the appropriate deposition method were experimentally determined.

2. Experimental methods

2.1. Sample preparation

In our experiment, the infrared coating samples were prepared with a coating plant (Leybold - SYRUSPro 1110) equipped with a diffusion-pump system, four boat evaporators and two electron beam sources. A quartz crystal oscillator was used to monitor the thickness and the deposition rate.

The infrared coatings were deposited on super-polished monocrystalline silicon substrates with 48 mm diameter and 2 mm thickness. Before deposition, the substrates were pre-cleaned with argon ion from APS ion source inside the vacuum chamber. During the deposition process, the pressure in the chamber was below 8×10^{-6} mbar. The samples of YbF_3 coatings with the thickness of approximately 1400 nm were deposited by either thermal evaporation with molybdenum boats or electron beam evaporation. For YbF_3 coatings, the deposition rates ranged from 0.16 nm/s to 0.74 nm/s, and the substrate temperature of YbF_3 coatings ranged from 60 °C to 240 °C. On the other hand, the samples of ZnS coatings with the thickness of approximately 840 nm were deposited by either thermal evaporation with

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Table 1
Defect density of YbF_3 coatings under different deposition process.

Sample no.	Substrate temperature (°C)	Deposition rate (nm/s)	Film thickness (nm)	Deposition method	Defect density (defects/mm^2)
Y1	60	0.38	1400	Mo-boat	8.1
Y2	100	0.38	1400	Mo-boat	4.2
Y3	150	0.38	1400	Mo-boat	3.1
Y4	200	0.38	1400	Mo-boat	32.2
Y5	240	0.38	1400	Mo-boat	>1000
Y6	100	0.37	1400	Mo-boat	4.6
Y7	100	0.16	1400	Mo-boat	96.5
Y8	100	0.74	1400	Mo-boat	6.4
Y9	150	0.38	1400	Electron beam	61.3

molybdenum boats or electron beam evaporation. For ZnS coatings, the deposition rates were in the range of 0.32 nm/s to 3 nm/s, and the substrate temperature ranged from 60 °C to 160 °C. The ZnS/YbF_3 multilayer coatings were 24-layer high-reflectance coatings.

2.2. Defect density measurements

A Leica optical microscope was used to count the numbers of defects on the coating samples. The defects were counted manually from 100× dark-field micrographs. Dark-field microscopy mode was chosen for defect detection instead of bright field microscopy mode due to its better sensitivity. During the course of the defect detection, dark dots detected by the observer were considered as defects in the coating samples. In this paper, the defect density is defined as the average defect density observed over twenty sites (micrograph) on each sample, that is, approximately 1.7% of the total sample area of 1809 mm^2.

3. Results and discussions

The measured defect density of YbF_3 coatings under different deposition process is shown in Table 1 and the defect density of ZnS coatings is given in Table 2.

Furthermore, the defect density of ZnS/YbF_3 multilayer coatings is also given in Table 3. The sample M1 was prepared at the beginning of the study and the sample M2 was prepared near the end of the study.

3.1. Deposition rate dependence of defect density

In general, when coatings are prepared at a low deposition rate, the residual particles within the coating chamber have more opportunity to adhere to the coatings because the deposition time is relative longer, resulting in much more defects in coatings. However, the molecules or atoms of the coating material have not enough time to deposit into coatings with non-defective structure when coatings are prepared at a high deposition rate. Furthermore, a high deposition rate results more probability of coating material spray. Both can cause more defects in coatings.

It can be seen from Table 1 that for YbF_3 single-layer samples, the optimum deposition rate is approximately 0.4 nm/s. Hence, when the deposition rate is below 0.2 nm/s, the defect density is increased by a factor of over 10 as compared to that under the optimum deposition rate. Furthermore, as is shown in Table 2 that the optimum deposition rate is approximately 1.2 nm/s for ZnS single-layer coatings.

When the deposition rate is low, the number of the defect becomes more while the size of the defect is small (usually a few micrometers). However, the number of the defect becomes less and the size of the defect is relative large (from a few micrometers to hundreds of micrometers) when the deposition rate is high. If the defect density is defined as the ratio of sum of defects area to area of samples, then the optimum deposition rate may be different, as described in reference [11].

3.2. Substrate temperature dependence of defect density

Substrate temperature is also an important factor that can cause defects in optical coatings during the deposition. Atomic adsorption and insufficient surface mobility of molecules caused by a number of factors enable nodule defects to grow. The low ratio of substrate temperature to the melting-point temperature of the coating material is a significant factor. In principle, a high substrate temperature can improve the atomic adsorption and surface mobility of molecules.

It can be seen from Table 1 that the defect density in YbF_3 coatings decreases with the increasing of the substrate temperature from 60 °C to 150 °C, which is consistent with the results in reference [12]. And the optimum substrate temperature is about from 100 °C to 150 °C. At this temperature range, the defect density in YbF_3 coatings is relative small. However, when the substrate temperature increases to above 200 °C, the microstructure of YbF_3 coatings is changed from amorphous state to crystalline state, resulting in much higher defect density. Fig. 1 shows the XRD patterns of the samples deposited under different substrate temperature (100 °C and 240 °C).

Table 2 shows that for ZnS coatings the optimum substrate temperature is from 80 °C to 120 °C. A lower or higher substrate temperature cause much more defect density. During the evaporation of ZnS coatings, the ZnS material does not melt into liquid but sublimate directly. Some ZnS molecules decompose into Zn atoms and S atoms. When Zn atoms and S atoms compose back into ZnS molecules again at the surface of the substrate, a higher substrate temperature can prevent Zn

Table 2
Defect density of ZnS coatings under different deposition process.

Sample no.	Substrate temperature (°C)	Deposition rate (nm/s)	Film thickness (nm)	Deposition method	Defect density (defects/mm^2)
Z1	60	1.2	840	Electron beam	57.1
Z2	80	1.2	840	Electron beam	38.3
Z3	100	1.2	840	Electron beam	15.9
Z4	120	1.2	840	Electron beam	24.1
Z5	160	1.2	840	Electron beam	85.8
Z6	100	0.32	840	Electron beam	73.1
Z7	100	0.61	840	Electron beam	58.8
Z8	100	0.92	840	Electron beam	22.9
Z9	100	1.2	840	Electron beam	15.2
Z10	100	1.7	840	Electron beam	20.1
Z11	100	0.61	840	Mo-boat	35.1
Z12	100	0.91	840	Mo-boat	17.3
Z13	100	1.2	840	Mo-boat	1.1
Z14	100	1.8	840	Mo-boat	1.7
Z15	100	3	840	Mo-boat	4.5
Z16	80	1.2	840	Mo-boat	3.2
Z17	80	1.2	840	Electron beam	35.2

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