Study on visible range beam splitter and 1570 nm high reflectivity film in a cemented cube

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We focus on a more difficult kind of beam splitter in a cemented cube. The specification is that when the incident angle is 45°, the reflectivity in the visible range is 70%±2%, and the reflectivity at 1570 nm is not less than 90%. The film is thick, so cracking and peeling often occur after coating without ion assisted deposition on a vacuum-coating plant made in China. Some experiments are conducted after changing the beam splitter's design, coating material, evaporation technique, and so on. Final results show that the beam splitter has good properties and can pass humidity, temperature, salt spray fog, and other kinds of environmental tests.

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The performance of modern military photoelectronic products and instruments has become more advanced and with more functions. Modern military photoelectronic systems are now equipped with a night vision meter, photoelectronic collimated tracker, laser ranger, charge-coupled device (CCD) camera, and other equipment. Miniaturization and light duty are known to be the developing trends. The beam splitter is one of the most important components in achieving small cubage and light weight properties of a photoelectronic instrument. In order to ensure the ideal performance of a photoelectronic system combining a visible wave band (450–656 nm) and laser wave band (1570 nm) in one body, it is necessary to fabricate a beam-splitting coating with a visible area energy spectrometer (R_{ave}:T_{ave} = 7:3) and a laser wave band with a high verse ($R \geq 90\%$).

With an incident angle of 45° in a cemented prism, the coating is relatively thick. If we fabricate this coating system without ion beam assistance in a China-made coating machine, the coating will easily crack. Moreover, military products have stricter requirements for environmental tests. The product is required to pass environmental tests, such as the high and low temperature test, damp heat test, and salt spray test. Therefore, we must manufacture the coating with a higher quality. In manufacturing this kind of beam-splitting coating, TiO_2 and SiO_2 were introduced. By conducting some research on coating design, stress observation, and coating techniques, the manufactured coating can meet the actual requirements.

The substrate is a prism glass BK7, and after it is cemented, the angle of incidence (AOI) is 45°. The specification is as follows

$$\begin{cases} \lambda_{01} = 450 - 656 \text{ nm}, \\ R_{\mathrm{ave}} : T_{\mathrm{ave}} = (70 \pm 2)\% : (30 \pm 2)\%, \\ \lambda_{01} = 1572 \text{ nm}, \ R \geq 90\% \end{cases}$$

The film structure used two modules of stack,

$$\mathrm{G}|(\mathrm{H}_1\mathrm{L}_1)^{S_1}(\mathrm{H}_2\mathrm{L}_2)^{S_2}\mathrm{H}_2 2\mathrm{L}_2|\mathrm{G}, \quad (1)$$

where G is the substrate (glass BK7), S_1 and S_2 are calculated by approximate expression $T_{\min} = 4(n_{\mathrm{g}}n_0/n_{\mathrm{H}}^2)(n_{\mathrm{L}}/n_{\mathrm{H}})^{2S}$, n_{g} is the refractive index of substrate (BK7), and n_0 is refractive index of incidence medium.

Under this condition, n_0 is also the refractive index of glass BK7.

Theoretically, as S value increases, the refrective index could reach 100% (Fig. 1). However, in actual situations, because of the absorbing and dispersion loss of the coating layer, when the coating system reaches a certain layer, the increased number of coatings hinders the increase in the reflection index. Sometimes, however, because of the increase in absorbing and dispersion loss, the reflection index could even decrease. Therefore, we must choose an appropriate S value[1].

If a periodical coating system is used, the coating layer will be too thick, and accordingly, the inner stress will surely increase. If an unperiodical coating structure is used, it will decrease the thickness and reduce the coating stress. An unperiodical coating system usually adopts an effective interface method (Smith pre-estimated strategies) for the design[2], the principle of which is to separate

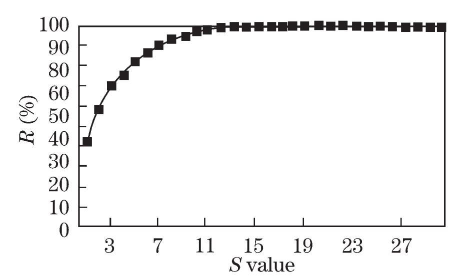

Fig. 1. Changing relationship between reflection index R and S value.

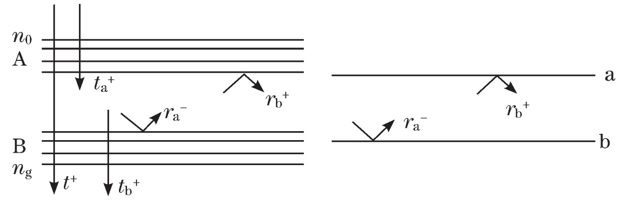

Fig. 2. Principle drawing of the effective interface method.

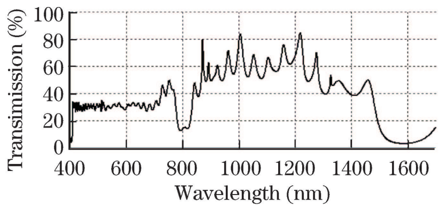

Fig. 3. Theoretical design.

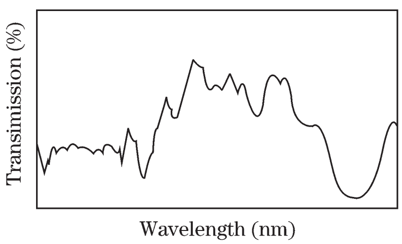

Fig. 4. Measurement results.

the selected coating layer from the coating structure, and thus, the whole coating structure can be shown by two effective interfaces. The characteristics of the coating structure can then be analyzed in consideration of the reflective coefficient between the two effective interfaces (Fig. 2).

According to the thin film theory, the transmittance of the coating structure can be calculated by the following formula

$$T = \frac{n_g}{n_o}(t^+)(t^+)^* = \frac{n_g t_a^+ t_a^{+*} t_b^+ t_a^{+*} e^{-2B}}{n_o(1 - t_a^- t_b^+ e^{-2B} e^{-2ia})(1 - r_a^- r_b^+ e^{-2B} e^{-2ic})^*}, \quad (2)$$

where t is transmission coefficient, r is reflection coefficient, + means the same direction of incidence, and - means the opposite direction of incidence.

Through this method, the best coating system can be obtained by optimizing the coating design software on a computer.

By suitably adjusting the H and L values of the initial coating system, after optimization, several sets of coating structures consisting of high and low refractive index materials with different proportions can be obtained. When we collocated the solution with high and low refractive coating materials at different rates, the coating system achieved minimum coating layer stress. The outmost $2L_2$ layer provides a good protection, which then allows the coating layer to successfully pass the environmental test[3]. The curve chart by theoretical design is shown in Fig. 3.

The coating was fabricated by a ZZS-700 coating machine (National Invested Nan Guang Co.). This machine was equipped with a molecular pump and two E-guns (Nanguang Co.). Rate and thickness were controlled by a quartz crystal monitor. However, the machine was not equipped with an ion source—this is an important feature, as the key technology needed to facilitate the coating process is the selection of a technique parameter that could decrease the coating stress. Coating stress includes surface tension, internal stress, and hot stress[4,5], and hot stress and internal stress are the main reasons of causing coating stress. Hot stress in the coating is mainly caused by different coefficients of heat expansion between the substrate and coating during the process of temperature decreasing to room temperature, this is unavoidable during the coating preparation process. Internal stress mainly depends on factors like the microstructure and defects of the coating. It mainly shows the following points during the coating process:

1) In the monolayer material test, we calculated each coating material's dispersion and absorption via the envelope method, and then designed the coating system in order to guarantee the optical performance.

2) TiO_2 coating and SiO_2 coating showed different stresses[6]. Various rates of TiO_2 and SiO_2 greatly influenced the coating stress. By conducting numerous experiments in selecting the coating system, we found that the coating system with a relatively thicker TiO_2 coating could hardly be split.

3) The assembled density of the film coating is important in determining internal stress. Along with the increase in assembled density, tensile stress could decrease accordingly and even cause press stress[7]. Since the hot stress of this coating system was tensile stress, the densification of the film coating decreased the tensile stress of the whole coating system. Since no ion source assistance exists, in order to enhance the assembled density, substrate temperature and evaporation speed were increased. Through testing, it was found that the higher the substrate temperature, the easier the coating split. As such, the substrate temperature was adjusted to 260 °C. To guarantee the surface quality of the coating, the evaporation speed rate was adjusted to 0.15 nm/s for TiO_2 and 0.2–0.3 nm/s for SiO_2, respectively.

4) When the oxygenate quantity was changed in order to improve the stress[8], it was found that too much oxygenate quantity could easily cause the coating to split. Under the condition wherein coating layer did not absorb, decreasing appropriately the oxygenate quantity showed great effect in controlling the stress. Through testing, we found that it was suitable to set the oxygenate quantity to 15 sccm. The vacuum rate after the oxygenate quantity process was retained at 1.5×10^{-2} Pa.

5) Upon completion of the coating, the high vacuum environment was maintained and the temperature was lowered step by step. The temperature was lowered by 30 °C every 30 min until the temperature was set to 80 °C. The coating was kept in the vacuum room overnight. The parts, which were taken out the next day, were annealed in the air. Firstly, the temperature was increased to 270 °C, maintained for 4 h, and then, it is lowered to room temperature for 10 h. After annealing, the optical performance almost remained the same. The annealing process greatly helped in solving the coating split.

The optical characteristics were measured by a 3101 spectrophotometer (SHIMADZU Co.). Measurement results are shown in Fig. 4.

Coating surface quality and mechanical abrasion, among others, were tested. An environmental test was conducted according to the following test standards:

1) Humidity test: The coating was kept for 24 h at a temperature of 40±2 °C and relative humidity of 90%–95%. The coating did not break off, and optical perfor-

mance met the requirements.

2) Salt spray test: The coating was salt-sprayed for 8 h with a concentration of 4.9%–5.1% and PH value of 6.5–7.2 at 35 °C. The coating did not break off, and optical performance met the requirements.

3) Low temperature test: The coating was kept at a low temperature of −40±3 °C for 2 h. The coating did not break off, and optical performance met the requirements. Meanwhile, the coating performance was good and likewise completely met the requirements.

In conclusion, we have got the following results:

1) The envelope method, which calculates material dispersion and absorption, could greatly ensure the optical performance, especially the dispersed part of the visible energy.

2) When using inhomogenous $\lambda/4$ coating, decreasing the thickness of the coating, and selecting the coating with the most appropriate assorted coating material could effectively decrease inner stress. The outmost $2L_2$ layer could greatly protect the coating and ensure that the coating layer passes the different environmental tests.

3) During coating, appropriately decreasing the oxygen and lowering the substrate temperature could effectively decrease coating stress.

4) Upon completion of the coating, the process of annealing in vacuum and air could effectively release the coating stress.

References

1. J. F. Tang and Q. Zheng, *Applied Thin Film Optics* (in Chinese) (Science Press Shanghai, Shanghai, 1980).
2. J. F. Tang, P. F. Gu, X. Liu, and H. F. Li, *Modem Optical Thin Film Technology* (in Chinese) (Zhejiang University Press, Hangzhou, 2006).
3. Y. X. Yan and H. H. Lin, *Thin Film Technology* (in Chinese) (The Publishing House of Ordnance Industry, Beijing, 1994).
4. M. F. Doerner, and W. D. Nix, Crit. Rev. Solid State Sci. **14,** 225 (1988).
5. H. K. Pulker, *Coating on Glasses* (Amsterdam: Elservier, 1984).
6. S. Xiong, Y. Zhang, and J. Tang, Opto-Electron. Eng. **28,** 1 (2001).
7. P. Gu, Z. Zheng, Y. Zhao, Acta Phys. Sin. **55,** 12 (2006).
8. D. Hao, Q. Wang, and L. Song, J. Optoelectron. Laser **20,** 5 (2009).