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Comparison of Measurement and Electromagnetic Overall Transfer Matrix Simulation Results of MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} Distributed Bragg Reflectors with Different Layers

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In this study, glasses were used as substrates and an e-beam was used the method to deposit MgF\textsubscript{2} and Nb\textsubscript{2}O\textsubscript{5} single-layer films, and the optical properties, including extinction coefficients (k values) and refractive indices (n values), were measured by using the light wavelength as variable. The equation $d = \lambda/(4n)$ was used to calculate the thickness (d) of 1/4 wavelength ($\lambda$) for each layer of the MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} bilayer films in distributed Bragg reflectors (DBRs) with a designed reflective wavelength at blue light (~450nm). Each MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} bilayer film was called a period, and the glass substrates were used to deposit the films with two, four, and six periods for fabricating the DBRs. The field emission scanning electron microscope equipped with a focused ion beam was used to measure the thickness of each MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} layer in the DBRs with different periods. The measured maximum reflective ratios were compared with Sheppard’s approximate equation, which calculates
only the maximum reflective ratio at a specific wavelength. An overall transfer matrix was investigated to calculate the reflective spectra by incorporating the variable n values and thicknesses of the MgF$_2$-Nb$_2$O$_5$ films in each layer. We show that the measured results of the fabricated DBRs matched the results simulated using Sheppard’s approximate equation and the overall transfer matrix.

Keywords: MgF$_2$-Nb$_2$O$_5$ bilayer film; Sheppard’s approximate equation; Overall transfer matrix

1. Introduction

Multilayer films are composite films deposited using numbers of alternating layers of different materials (for example, metal oxides), these films have their specific thicknesses as they are with nanometer scale. Multilayer films with different materials have been widely used in a large number of applications because they have outstanding optical, photoactive, and protective properties. For example, to achieve a long lifetime for semiconductive lasers with a microcavity exciton-polariton, it is necessary to use a highly reflective distributed Bragg reflector (DBR)$^{1,2}$. A DBR is a reflector designed for a specific wavelength with a narrow bandwidth, which can be used to design a notch filter$^3$. DBRs are also used in waveguides, and they can be constructed from multiple layers of different materials with periodic variations in refractive indices (n values, materials with low and high n values are alternately stacked in the vertical direction), and these films have a quarter-wave ($\lambda$/4) thickness of the designed reflective wavelength. Bilayer film designs can be found in commercial wavelength division multiplexers, which have single-period structures of H(Ta$_2$O$_5$/L(SiO$_2$), H(Nb$_2$O$_5$/L(SiO$_2$), H(TiO$_2$/L(SiO$_2$), or H(TiO$_2$/L(Al$_2$O$_3$) and use different numbers of periods$^{4,6}$.

Many materials have been used to form the periodically varying layers, including organic polymers and inorganic oxides$^7$. Guldin et al. investigated a fast and versatile route to fabricate mesoporous DBRs. They used the self-assembling isoprene-\textit{block}-ethylene oxide (PI-\textit{b}-PEO) in two stack solutions containing PI-\textit{b}-PEO copolymer and TiO$_2$ sol with different weight ratios to sequentially spin-cast layers, forming stacks of alternating compositions$^8$. Many deposition technologies have been investigated to deposit films with specific requirements or properties. For example, Lin et al. used a spin-coating and annealing technique to deposit PbZr$_{0.4}$Ti$_{0.6}$O$_3$ multilayer
films\textsuperscript{9}, and Wang et al. used a CO\textsubscript{2} laser to fabricate SiO\textsubscript{2}-Al\textsubscript{2}O\textsubscript{3} glasses with a thermally stable n value\textsuperscript{10}. Dubey and Ganesan used a sol-gel spin-coating technique to fabricate DBRs based on TiO\textsubscript{2}-SiO\textsubscript{2} stacks, but as the TiO\textsubscript{2}-SiO\textsubscript{2} films were deposited in seven periods, only about 90\% reflectance was observed\textsuperscript{11}. Muallem et al. used a thermal evaporation method to fabricate multilayer DBRs using CaF\textsubscript{2} as material with lower n value and ZnS as material with higher n value, and they found that the fabricated DBRs exhibited a center wavelength of 550nm with a maximum reflectance more than 99\%\textsuperscript{12}.

Willey and Shakoury used ion beam-assisted deposition to deposit an MgF\textsubscript{2} film without additional heat or fluorine, and the deposited film exhibited low absorbance, low scattering, and a low n value\textsuperscript{13}. Chen et al. used a sputtering method to deposit Nb\textsubscript{2}O\textsubscript{5} films under different oxygen concentrations and found that when they introduced oxygen during the deposition process, the deposited Nb\textsubscript{2}O\textsubscript{5} film had low absorbance and a high n value\textsuperscript{14}. In the present study, we used the MgF\textsubscript{2} and Nb\textsubscript{2}O\textsubscript{5} films as low and high refractive-index films to fabricate a DBR with a centrally reflective frequency of 450nm. An electron-beam (e-beam) deposition can be a simple method to deposit the different oxide films, and the properties of the deposited films depend on the deposition parameters, such as temperature and pressure, and the used gas during deposition\textsuperscript{5,6,14}. The e-beam deposition method makes it easy to control the thicknesses of the films, even in multilayer structures or special structures, so the e-beam technology was used for deposition the MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} bilayer films.

In the past, Du et al. also used bi-layer MgF\textsubscript{2} and Nb\textsubscript{2}O\textsubscript{5} films to design a 500 nm DBR, but they only used the e-beam to deposit the bi-layer MgF\textsubscript{2} and Nb\textsubscript{2}O\textsubscript{5} films and investigated their properties\textsuperscript{15}. In this study, we would investigate a transfer matrix to find the difference between the simulated and measured results of the fabricated DBRs. The variations in the reflective ratios of multilayer MgF\textsubscript{2}-Nb\textsubscript{2}O\textsubscript{5} DBRs versus the variations in period numbers is simulated using Sheppard’s approximate equation\textsuperscript{16}:

\[
R = \frac{\left(\frac{n_H}{n_L}\right)^{2P} - \left(\frac{n_0}{n_S}\right) \times \left(\frac{n_L}{n_H}\right)^{2P}}{\left(\frac{n_H}{n_L}\right)^{2P} + \left(\frac{n_0}{n_S}\right) \times \left(\frac{n_L}{n_H}\right)^{2P}}
\]

(1)

where \(n_L\), \(n_H\), \(n_S\), and \(n_0\) are MgF\textsubscript{2} (\(n_L = 1.39\)), Nb\textsubscript{2}O\textsubscript{5} (\(n_H = 2.29\)), glass substrate (\(n_S = 1.52\)), and air.
(n₀ = 1), R represents the reflective ratio at a special wavelength of light, and P is the deposited period of the MgF₂-Nb₂O₅ bilayer films.

If the n values of the two films differ greatly, the fabricated DBR will have a similar reflective ratio if fewer periods are deposited, or a higher reflective ratio if the designed device has the same number of periods. Knowing this saves time and money when designing DBRs, due to the n values of the MgF₂ (n₁= 1.39) and Nb₂O₅ films (n₃ =2.29). MgF₂ and Nb₂O₅ films have low extinction coefficients (k values), as well as high transparent ratios in the near-infrared, visible, and ultraviolet regions. In optical applications, Nb₂O₅ film can be used as a low-loss and high-index material, and MgF₂ film can be used as a low-loss and low-index material. There are two reasons for us to use MgF₂ and Nb₂O₅ films to design reflective films having a multi-period L (MgF₂, 1/4 λ)/H (Nb₂O₅, 1/4 λ) bilayer structure. The first is that the MgF₂ film (n ~1.39 in light range of 400~700nm)¹⁷ and the Nb₂O₅ film (n ~2.30 in visible light)¹⁴ have very different n values. The second is that both materials have low k values¹⁴,¹⁷. In this paper, we show that even with only four periods of the MgF₂-Nb₂O₅ bilayer films deposited, the fabricated DBRs had a reflective ratio of over 90%.

However, Equation (1) is only used to simulate the maximum reflective ratio at a designed wavelength, and it cannot simulate the reflective spectrum in a very wide range of light wavelength. In this study, we investigated the transfer matrix of each layer and obtained an overall transfer matrix of the multilayer coatings, which is formed using the product of each single transfer matrix. The following steps were carried out for investigating the potential difference between the experimental measurements and simulation estimations for an MgF₂-Nb₂O₅ bilayer blue-light Bragg reflector with different periods. First, we deposited single-layer MgF₂ and Nb₂O₅ films on glass substrates, their k values and n values were measured, and the thicknesses to match the λ/4 MgF₂ and Nb₂O₅ films could be obtained. Next, a characteristic matrix for simulating the multilayer films was constructed to calculate the reflective spectra of a bilayer MgF₂-Nb₂O₅ Bragg reflector with different periods by using the optical properties of the single-layer MgF₂ and Nb₂O₅ films. Third, we deposited bilayer MgF₂-Nb₂O₅ DBRs according to the calculated thicknesses of the λ/4 MgF₂ and Nb₂O₅ films, then measured the reflective spectra of the fabricated DBRs. In what follows, we provide a detailed
comparison of the experimental and theoretical results for the multilayer blue-light MgF$_2$-Nb$_2$O$_5$ DBRs and discuss reasons for deviations between the measured and simulated results.

2. Experimental Procedure

Corning 1737 glass substrates (the area was $2 \times 2$ cm$^2$) were cleaned, then MgF$_2$ and Nb$_2$O$_5$ single-layer films were deposited on them using e-beam technology to measure their $k$ values and $n$ values. The thickness of the Corning 1737 glass was about 2 mm. The deposited MgF$_2$ and Nb$_2$O$_5$ single-layer films and the MgF$_2$-Nb$_2$O$_5$ bilayer films were much thinner than the glass substrates, to prevent the coherent condition that would be caused by multiple light reflections in the substrates. The base chamber pressure to start the deposition was $6 \times 10^{-6}$ Torr, for the electron beam the applied voltage and current were 4 kV and 20 mA, and 25°C (room temperature) was used the deposition temperatures of the MgF$_2$ and Nb$_2$O$_5$ films. The thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films were 57.7 and 58.4 nm, which were controlled by a film gauge on the e-beam and verified by field-emission scanning electron microscopy (FESEM). When the MgF$_2$ and Nb$_2$O$_5$ films were deposited at higher temperatures or were annealed, the films' densities increased and/or the average crystallite sizes increased. There were two important reasons for us to deposit the MgF$_2$ and Nb$_2$O$_5$ films at room temperature. First, as the films’ densities increased, their thicknesses decreased and became not easy to control, causing variations in the central wavelength of the reflective band. Second, as the crystallite sizes increased, grain boundaries formed between crystallites of different sizes; this increased the chance of the light scattering, which in turn affected the reflectance and transmittance efficiencies of the multilayer films.

For the deposition of the MgF$_2$-Nb$_2$O$_5$ bilayer films, at first an MgF$_2$ film was deposited on a glass substrate, after that a Nb$_2$O$_5$ film was deposited on the MgF$_2$ film, and then one period of the MgF$_2$-Nb$_2$O$_5$ bilayer film was formed, as Figure 1 shows. For the investigation of the effect of period number on the properties of the designed blue-light DBRs, different periods of the MgF$_2$-Nb$_2$O$_5$ bilayer films were deposited. The DBRs’ center wavelength was 450 nm, and the thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films were 80.9 and 49.0 nm, respectively. The $k$ values and $n$ values of the MgF$_2$
and Nb$_2$O$_5$ films were measured along the direction of thickness using a normal light incidence on single-layer films with an n&k analyzer. The n&k analyzer was able to measure thickness (d), n value, k value, transmittance spectrum, and reflectance spectrum, and it used a nonlinear curve-fitting method, which fit the measured results using polynomial terms in linear regression, and these measured data can be retrieved directly. X-ray diffraction (XRD) pattern was used to measure the crystalline structure of the MgF$_2$ and Nb$_2$O$_5$ single-layer films and the MgF$_2$-Nb$_2$O$_5$ bilayer film with different periods. The surface observations of top-layer films for different periods of MgF$_2$-Nb$_2$O$_5$ bilayer film were measured using FESEM. For measuring the real thicknesses of each MgF$_2$ and Nb$_2$O$_5$ film for different periods, we used a focused ion beam to prepare the samples and used the FESEM to observe the cross sections of the MgF$_2$-Nb$_2$O$_5$ bilayer films. The n&k analyzer was also used to measure the reflective spectra of the MgF$_2$-Nb$_2$O$_5$ bilayer films with different periods in the light wavelength range of 300–800nm.

**Figure 1.** Structure of designed MgF$_2$-Nb$_2$O$_5$ bilayer films with different periods.

Light is one kind of electromagnetic wave, for a single-layer film on a substrate (substrate-thin film-air), the interface between substrate and film and that between film and air were denoted by b and a, respectively, and the transmission (τ) and amplitude reflection (ρ) coefficients were$^{18,19}$:

$$\tau = \frac{E_b}{E_{ba}^0} = \frac{2\eta_0 E_b}{\eta_0 E_a + H_a}, \quad \rho = \frac{E_{ba}^0}{E_{ba}^0} = \frac{\eta_0 E_a - H_a}{\eta_0 E_a + H_a}$$

(2)

Therefore, the relationship between magnetic field ($H_a$) and electrical field ($E_a$) can be written in matrix equation shown below$^{18,19}$:

$$\begin{bmatrix} E_a \\ H_a \end{bmatrix} = \begin{bmatrix} \cos\delta & \frac{i}{N} \sin\delta \\ iN\sin\delta & \cos\delta \end{bmatrix} \begin{bmatrix} E_b \\ H_b \end{bmatrix}$$

(3)
where $\delta$ represents the phase factor and it can be shown below

$$\delta = 2\pi N d \cos(\theta)/\lambda \quad (4)$$

Where $N$ is the complex index and it is equal to $(n_r - ikr)$, $d$ is the thickness of the film, $\lambda$ is the wavelength of light, $k$ is the extinction coefficient, and $n$ is the refractive index. $N$ is a modified optical admittance of the deposited film and it can be shown as below:

$$N = \frac{E}{H} \quad (5)$$

For the multilayer films, when light is propagated from the front face of a reference plane (often is the air) to the surfaces of the multilayer films, and then to the substrate, the variation in the input optical admittance can be listed as follows for single-layer, two-layer (one period, $P=1$), four-layer (two periods, $P=2$), eight-layer (four periods, $P=4$), and twelve-layer (six periods, $P=6$) structures:

$$\begin{bmatrix} B_1 \\ C_1 \end{bmatrix} = \begin{bmatrix} \cos\delta_1 \\ iN\sin\delta_1 \\ iN\sin\delta_1 \end{bmatrix} \begin{bmatrix} \frac{i}{N} \sin\delta_1 \\ \cos\delta_1 \end{bmatrix} \begin{bmatrix} 1 \\ Y_s \end{bmatrix}, \quad Y_1 = \frac{B_1}{C_1}$$

$$\begin{bmatrix} B_2 \\ C_2 \end{bmatrix} = \begin{bmatrix} \cos\delta_2 \\ iN\sin\delta_2 \\ iN\sin\delta_2 \end{bmatrix} \begin{bmatrix} \frac{i}{N} \sin\delta_2 \\ \cos\delta_2 \end{bmatrix} \begin{bmatrix} 1 \\ Y_1 \end{bmatrix}, \quad Y_2 = \frac{B_2}{C_2}$$

$$\begin{bmatrix} B_4 \\ C_4 \end{bmatrix} = \begin{bmatrix} \cos\delta_4 \\ iN\sin\delta_4 \\ iN\sin\delta_4 \end{bmatrix} \begin{bmatrix} \frac{i}{N} \sin\delta_4 \\ \cos\delta_4 \end{bmatrix} \begin{bmatrix} 1 \\ Y_3 \end{bmatrix}, \quad Y_4 = \frac{B_4}{C_4}$$

$$\begin{bmatrix} B_8 \\ C_8 \end{bmatrix} = \begin{bmatrix} \cos\delta_8 \\ iN\sin\delta_8 \\ iN\sin\delta_8 \end{bmatrix} \begin{bmatrix} \frac{i}{N} \sin\delta_8 \\ \cos\delta_8 \end{bmatrix} \begin{bmatrix} 1 \\ Y_9 \end{bmatrix}, \quad Y_8 = \frac{B_8}{C_8}$$

$$\begin{bmatrix} B_{12} \\ C_{12} \end{bmatrix} = \begin{bmatrix} \cos\delta_{12} \\ iN\sin\delta_{12} \\ iN\sin\delta_{12} \end{bmatrix} \begin{bmatrix} \frac{i}{N} \sin\delta_{12} \\ \cos\delta_{12} \end{bmatrix} \begin{bmatrix} 1 \\ Y_{11} \end{bmatrix}, \quad Y_{12} = \frac{B_{12}}{C_{12}}$$

$$R = |\rho|^2 = \frac{|Y_0 - Y_4|^2}{|Y_0 + Y_4|^2} \quad \text{for } P = 2$$

$$R = |\rho|^2 = \frac{|Y_0 - Y_8|^2}{|Y_0 + Y_8|^2} \quad \text{for } P = 4$$

$$R = |\rho|^2 = \frac{|Y_0 - Y_{12}|^2}{|Y_0 + Y_{12}|^2} \quad \text{for } P = 6$$

where $Y_s$ is the admittance of the substrate.

### 3. Results and Discussion

At first, we used XRD patterns to measure the crystalline structures of the MgF$_2$ and Nb$_2$O$_5$ single-layer films and one-period MgF$_2$-Nb$_2$O$_5$ bilayer films; the results are shown in Figure 2. No characteristic peaks were found in the XRD patterns, because these films deposited using the e-beam
process would present in the amorphous phase. We believe that because the e-beam deposition process was carried out at room temperature, the natures of the single-layer and bilayer films remained unchanged. The XRD patterns of MgF$_2$-Nb$_2$O$_5$ bilayer films with different periods were also measured (not shown here); again, only the amorphous phase was observed.

![XRD patterns](image)

Figure 2. XRD patterns of MgF$_2$ and Nb$_2$O$_5$ single-layer films and MgF$_2$-Nb$_2$O$_5$ bilayer film.

In optics, the $n$ value of a film or material is a unitless number, which describes how the light or electromagnetic wave propagates into or through the film or material. The $n$ value can be defined as $n = c/v$, where $v$ is the speed of light in the film or material and $c$ is the speed of light in a vacuum. The $n$ value is not a stable value and may change as the incident light changes its wavelength. The $k$ value, also called the molar extinction coefficient, is a measure of how much a chemical species or material absorbs incident light at a particular wavelength. It is an intrinsic property and is strongly dependent upon the species or material’s composition. The $n$ and $k$ values are corresponding to the interaction between incident light and a film or material, and the $k$ value is associated with refraction and absorption.

The $n$ values and $k$ values of the MgF$_2$ film and glass substrate were measured in the light wavelength range of 200–1700nm. However, the optical properties of the Nb$_2$O$_5$ film were measured in the light wavelength range of 350–1700nm, because as the light wavelength was shorter than 350nm, its $n$ and $k$ values increased sharply, and correct values were difficult to obtain. The $n$ values of the MgF$_2$ and Nb$_2$O$_5$ films and the glass substrate were measured as a function of light wavelength.
and the results are shown in Figure 3(a). The Nb$_2$O$_5$ film had an n value of 2.52 at 350nm, which decreased sharply as the light wavelength became longer; at 400, 500, and 700nm, the n values were 2.38, 2.23, and 2.15, respectively. The n value of the Nb$_2$O$_5$ film underwent no apparent change when the light wavelength was longer than 700nm. The MgF$_2$ film (glass substrate) had an n value of 1.45 (1.64) at 200nm, the n value decreased slightly as the light wavelength became longer, and at 300 and 400nm, the n values were 1.41 (1.55) and 1.40 (1.52). In this case, the n values of the MgF$_2$ film and glass substrate remained presenting the stable values as the light wavelength increased. Because we wanted to fabricate an optical reflector with a central wavelength of blue light, we chose n values of 1.39 and 2.29 (both at 450nm) for the MgF$_2$ and Nb$_2$O$_5$ films, using the equation $d = \lambda/(4n)$ to calculate the thicknesses that matched the designed wavelength of quarter wave, where n is the refractive index, $\lambda$ is the targeted wavelength of 450nm, and d is the film thickness. The thicknesses of 80.9 and 49.0nm were matched the thicknesses of MgF$_2$ and Nb$_2$O$_5$ films for the $\lambda/4$ condition.

Figure 3(b) shows the k values of the glass substrate and the Nb$_2$O$_5$ and MgF$_2$ films in a spectrum range of 200–1700nm. As the light wavelength increased from 200 to 300nm, the k value of the glass substrate decreased quickly from ~0.018 to close to zero. When the light wavelength increased from 350 to 650nm, the k value of the Nb$_2$O$_5$ single-layer film decreased quickly from ~3.6% to ~1.6%, and the k value was less than 1% when the light wavelength was longer than 1200nm. Previously, Chen et al. used a sputtering method to deposit Nb$_2$O$_5$ films in different atmospheres$^{[14]}$. They found that when pure argon was used as the deposition atmosphere, the Nb$_2$O$_5$ film had a higher k value; when oxygen was injected with the argon during the deposition, the Nb$_2$O$_5$ film had a lower k value, which dropped to zero as the light wavelength was longer than 346–370nm.

This was because when pure argon was used, a small amount of Nb$_2$O$_5$ decomposed to form Nb$_2$O$_{5-x}$, which had semiconducting characteristics and thus increased the k value. When oxygen was mixed with the argon, the Nb$_2$O$_5$ film did not decompose to form the Nb$_2$O$_{5-x}$ and had a lower k value. Because we deposited the Nb$_2$O$_5$ film in a high vacuum, we believe that only a small amount of the Nb$_2$O$_5$ decomposed to form Nb$_2$O$_{5-x}$, creating oxygen vacancies in the film. The oxygen vacancies increased the film’s conductivity and absorption, so the Nb$_2$O$_5$ (Nb$_2$O$_{5-x}$) film had semiconducting
properties and a higher k value. However, the deposited MgF$_2$ film had a k value close to zero even when the measured wavelength was 200nm. This result suggested that the deposited MgF$_2$ film was a stable and lossless dielectric material. The results in Figure 3(b) suggest that the glass substrate and the Nb$_2$O$_5$ and MgF$_2$ films were low-loss dielectric materials because they had low k values in the range of visible light, so they could be used to fabricate DBRs with reflective wavelengths in the visible light spectrum.

Cauchy had proposed a transmission equation to express an empirical relationship between the n value and light wavelength for a transparent film or material$^{20}$. The equation is valid for the region of normal dispersion in the visible wavelength range. The most general form of Cauchy’s equation is

$$n(\lambda) = a + \frac{b}{\lambda^2} + \frac{c}{\lambda^4} + ....$$                                             (12)

where $n$ is the n value of the used transparent material; $\lambda$ is the measured wavelength; and $a$, $b$, $c$, etc. are coefficients, which are used for determining the parameters in the equation for a material to fit the variables of the n values at a measured wavelength. The coefficients are usually quoted for $\lambda$ as the vacuum wavelength in a material, measured in μm. Typically, using a three-term form of the equation is sufficient to express a material:

$$n(\lambda) = a + \frac{b}{\lambda^2} + \frac{c}{\lambda^4}$$                                             (13)

For the MgF$_2$ film, the a, b, and c values are 1.3805820, 2.3938716 $10^{-3}$ (μm$^2$), and 1.6955936 x $10^{-5}$ (μm$^4$), and for the Nb$_2$O$_5$ film, the a, b, and c values are 2.0830535, 2.6614043 $10^{-2}$ (μm$^2$), and 3.2799308 x $10^{-3}$ (μm$^4$). Apparently, as Figures 3(c) and 3(d) show, the n values calculated using Equation (12) match the variations in the Nb$_2$O$_5$ and MgF$_2$ films with different wavelengths. Those results have proven that the decreases in the n values of the Nb$_2$O$_5$ and MgF$_2$ films were caused by their normal dispersion.
Figure 3. Measured (a) n values and (b) k values of glass substrate and single-layer MgF$_2$ and Nb$_2$O$_5$ films; (c) and (d) K values of single-layer MgF$_2$ and Nb$_2$O$_5$ films compared with Cauchy equation.

After the thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films matched for the $\lambda/4$ condition were found, the MgF$_2$-Nb$_2$O$_5$ bilayer films with the periods of two, four, and six were deposited using the e-beam process. The surface morphologies in Figure 4 compares the surface morphologies of the bilayer films. These FESEM images show that when the deposition was done at room temperature, a dense surface morphology was presented on the surfaces of Nb$_2$O$_5$ films, consisting of growing nanocrystalline particles, which were polycrystalline materials with a grain size of only a few nanometers. Although nanocrystalline particles were observed, the XRD patterns indicated that the MgF$_2$ and Nb$_2$O$_5$ single-layer films and the MgF$_2$-Nb$_2$O$_5$ bilayer films presented amorphous structures. The results in Figure 4 also show that the nanocrystalline Nb$_2$O$_5$ grains presented the uniform particle sizes. The average crystallite sizes were 12.3, 16.7, and 22.1nm for two (Figure 4(a)), four (Figure 4(b)), and six (Figure 4(c)) periods.

Figure 4. Surface morphologies of MgF$_2$-Nb$_2$O$_5$ bilayer films with (a) two, (b) four, and (c) six periods.
An AFM with a tapping mode could be used to analyze the surfaces’ roughness of the Nb₂O₅ films on top of the MgF₂-Nb₂O₅ bilayer films with different period numbers. Because we deposited the MgF₂-Nb₂O₅ bilayer films at room temperature, we believed they would have a lower activation energy, and the Nb₂O₅ films would have smaller particle sizes and less roughness. As two, four, and six periods were deposited, as Figure 5 shows, their average roughness (Sa) was 1.21, 1.28, and 1.35nm and their root-mean-square (rms, Sq) surface roughness of the Nb₂O₅ films was 1.56, 1.68, and 1.75nm. These results mean that the roughness of the Nb₂O₅ films on four- and six-period bilayers was greater than on two periods. The results also prove that all the Nb₂O₅ films’ surfaces were relatively smooth, but their roughness slightly increased with the period number.

Figure 5. Roughness analysis of MgF₂-Nb₂O₅ bilayer films with (a) two, (b) four, and (c) six periods.

Because we wanted to fabricate a DBR for reflecting the blue light and with a central wavelength of 450nm, the n values of 1.29 and 2.29 were incorporated into the equation \( d = \frac{\lambda}{4n} \) to determine the thicknesses (d) of the single-layer MgF₂ and Nb₂O₅ films. The calculated thicknesses of the MgF₂ and Nb₂O₅ films for each period were 80.9 and 49.0nm, and the calculated total thicknesses of the MgF₂-Nb₂O₅ bilayer films with two, four, and six periods were 259.8, 519.6, and 779.4nm. FESEM cross-sectional images of the DRBs with different periods are shown in Figure 6, where the entire multilayer structure can be seen. The total thicknesses of the deposited MgF₂-Nb₂O₅ bilayer films with two, four, and six periods were 242.8, 520.0, and 780.5nm. The average thicknesses of the MgF₂ films for two, four, and six periods were 76.7 (75.3–78.1), 81.0 (80.3–81.5), and 81.3nm (80.2–82.2),
and those of the Nb$_2$O$_5$ films were 44.6 (44.0–45.2), 49.0 (48.4–49.7), and 48.8nm (48.4–49.4).

The thicknesses of the individual MgF$_2$ and Nb$_2$O$_5$ films in Figure 6 have only minor deviations and are in agreement the thicknesses with the target values of d = 80.9 and 49.0nm, as obtained from the theoretical calculations using d = λ/(4n). These cross-sectional images show that no voids were formed in the MgF$_2$-Nb$_2$O$_5$ bilayer films, and no boundary defects or grain growth could be found at the interfaces of each MgF$_2$ and Nb$_2$O$_5$ films. Because no small particles or voids were formed and no boundary defects or grain growth were generated at the interfaces of each MgF$_2$ and Nb$_2$O$_5$ films, we were able to disregard Mie and Rayleigh scatterings$^{21}$ and identify the MgF$_2$-Nb$_2$O$_5$ bilayer films with different periods as homogeneous films with uniform n values.
Figure 6. Cross-sectional observations of MgF$_2$-Nb$_2$O$_5$ bilayer films with (a) two, (b) four, and (c) six periods.

To further confirm the compositions of each MgF$_2$ and Nb$_2$O$_5$ layers, we used energy-dispersive X-ray (EDX) to perform the analyses on the cross-sectional structures. EDX analyses proved the presence of elemental Mg and F within the MgF$_2$ layer and elemental Nb within the Nb$_2$O$_5$ layer. Figure 7 shows the dispersions of the elements in each MgF$_2$ and Nb$_2$O$_5$ layers using the EDX mapping analysis. The dispersions confirm that the multilayer films were indeed composed of MgF$_2$ and Nb$_2$O$_5$, and good separation between each MgF$_2$ and Nb$_2$O$_5$ layers were also really observed. The elemental ratios for Mg, F, Nb, and O were analyzed using FESEM equipped with EDX, and the average atomic ratio of Mg to F was 2.00 (±0.015):1 and that of Nb to O was 2.51 (±0.018):1. Another important reason affecting the reflective properties was that the MgF$_2$ and Nb$_2$O$_5$ films formed compositions when the MgF$_2$ and Nb$_2$O$_5$ films were deposited. Because the films were deposited at room temperature, we can observe in the Figure 7 cross-sectional images that there were boundaries in the different MgF$_2$ and Nb$_2$O$_5$ layers. The properties of central wavelength and reflective ratio in the reflective spectra matched those of the simulation results, indicating a reaction between the MgF$_2$ and Nb$_2$O$_5$ films did not happen.
Figure 7. Cross-sectional EDX mapping measurements of four-period MgF$_2$-Nb$_2$O$_5$ bilayer films.

After the two, four, and six periods of MgF$_2$-Nb$_2$O$_5$ bilayer films were deposited, the optimum reflective ratios of optical spectra were 66.8%, 92.5%, and 95.3% at light wavelengths of 436nm, 456nm, and 448nm. Using Equation (1), the maximum reflective ratios were calculated to be 70.0%, 95.3%, and 99.3% when the period numbers of the designed DBRs were two, four, and six. When these simulation results are compared with the measured values, they have no apparent differences in the central light wavelength. The calculated bandwidth for the reflective band of the fabricated DBRs with the MgF$_2$-Nb$_2$O$_5$ bilayer films at a specific wavelength (λ) can be found using Equation (14):

$$\frac{\Delta \lambda}{\lambda} = (\frac{4}{\pi}) \arcsin \left[ \frac{n_H - n_L}{n_H + n_L} \right]$$ (14)

where $n_L$ and $n_H$ are the n values of the MgF$_2$ and Nb$_2$O$_5$ films, $\lambda$ is the central light wavelength for reflectance of the designed DBR, and $\Delta \lambda$ is the reflective bandwidth of DBRs at a specific wavelength. When the measured n values of the MgF$_2$ ($n_L = 1.39$) and Nb$_2$O$_5$ ($n_H = 2.29$) films are used in Equation (13) to find the bandwidth at the stop-band of blue light (450nm) for the MgF$_2$-Nb$_2$O$_5$ bilayer films, the calculated value is 142nm, meaning light in the light wavelength of 379–521nm can be reflected. The measured reflective bandwidths and simulated reflective bandwidths (using an overall transfer matrix) are defined as that the measured reflective ratio is higher than or equal to 90% of the measured maximum reflective ratio. Hence, as Figure 8 shows, for the bilayer films with two, four, or six
periods their measured bandwidths were 88nm (385–497nm), 113nm (409–522nm), and 116nm (395–
511nm). The full width at half maximum (FWHM) values of the reflected bands with two, four, or
six periods were 263nm (347–610nm), 186nm (386–572nm), and 160nm (384–544nm). These results
prove that the simulated results have matched the measured ones and also prove that differences in
thicknesses and the n values of the MgF$_2$ and Nb$_2$O$_5$ films are the reasons for the variation in the
central light wavelength and the difference in bandwidth.

Figure 8. Measured reflectance rates of MgF$_2$-Nb$_2$O$_5$ bilayer films as a function of period number.

Figure 9 presents the measured transmittance ratio for the MgF$_2$-Nb$_2$O$_5$ bilayer films with different
period numbers. Figures 8 and 9 show an important result: when the light wavelengths of
transmittance and reflective spectra are longer than 350nm, the reflective ratio and the transmittance
ratio have inverse results, and the addition of both values is almost equal to 1.
Figure 9. Measured transmittance rate of MgF$_2$-Nb$_2$O$_5$ bilayer films with different period numbers.

The reflectance spectra of the experimental measurements and the theoretical calculations of the MgF$_2$-Nb$_2$O$_5$ bilayer films in the light wavelength of 300–800nm are compared in Figure 10. The theoretical results were calculated by incorporating the variable $n$ values and thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films into the calculations for the overall transfer matrices (Equations (6)-(9)). As Figure 10 shows, the experimental reflectance spectra were similar to the simulation results in the light wavelength of 300–800nm, and the measured central wavelengths for the three designed reflectors overlapped with the simulation results. This outcome suggests that the theoretically calculated spectra have good agreement with the experimentally measured results when we used the variable thicknesses of each layer and the variable $n$ values of each wavelength for the whole MgF$_2$-Nb$_2$O$_5$ bilayer films. Figure 10 indicates that when the wavelength was larger than 500nm, moderate deviations in the reflective spectra were observed for the MgF$_2$-Nb$_2$O$_5$ bilayer films with different periods.

Many reasons can cause the designed DBRs with different-period MgF$_2$-Nb$_2$O$_5$ bilayer films having the deviations in their central light wavelengths. The two main theorems could have been variations in the thicknesses of the MgF$_2$-Nb$_2$O$_5$ bilayer films and decreases in the $n$ values of the MgF$_2$ and Nb$_2$O$_5$ films as the light wavelength increased. We believe that differences in the individual and total thicknesses of the multilayer MgF$_2$-Nb$_2$O$_5$ films caused the small differences between the simulated (obtained from $d=\lambda/(4n)$) and reflective central wavelengths and reflectance bandwidths. The FESEM images in Figure 6 were used to measure the thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films.
in each layers, and the average thicknesses of the MgF$_2$ and Nb$_2$O$_5$ films for the deposited DBRs with two periods were 76.7 and 44.6nm. As using the equation of $d = \lambda/(4n)$, the central light wavelengths determined from the MgF$_2$ and Nb$_2$O$_5$ films having the thicknesses of 76.7 and 44.6nm were 427 and 410nm. Although the two values are close to the measured and simulated results of 439 and 434nm, they still caused deviations in the central wavelengths because of the deviations in the films’ thicknesses.

The thicknesses of each of the MgF$_2$ and Nb$_2$O$_5$ films in the fabricated DBRs with four and six periods were also measured, their average thicknesses in the bilayer films with four periods were 81.0 and 49.0nm, and with six periods were 81.15 and 48.85nm. From the equation of $d = \lambda/(4n)$, the central light wavelengths for the MgF$_2$ and Nb$_2$O$_5$ films with thicknesses of 81.0 and 49.0nm were 450 and 450nm, and for the MgF$_2$ and Nb$_2$O$_5$ films with thicknesses of 81.15 and 48.85nm their respectively central light wavelengths were 451 and 449nm. These values were close to the measured and simulated results of 456 and 448nm for four periods and of 459 and 439nm for six periods. As Figure 6 shows, the thicknesses of the individual MgF$_2$ and Nb$_2$O$_5$ films in different periods were different, so their central light wavelengths for each layer were also different. We believe that deviations in the thicknesses of the fabricated DBRs are the main reason for the shift in their central light wavelengths, and the results obtained from the simulation of the investigated overall transfer matrix prove this to be the case. As the periods of the bilayer films were two, four, and six, their simulated bandwidths were 103nm (390–493nm), 140nm (400–540nm), and 104nm (398–502nm). However, the bandwidths for these bilayer films with the same periods and simulated with an overall transfer matrix matched the measured results.
Figure 10. Simulated and measured reflectance spectra of DBRs designed using the MgF$_2$-Nb$_2$O$_5$ bilayer films: (a) two, (b) four, and (c) six periods.

Figure 11 compares the maximum reflective ratios using the two simulation methods, and the measured results for the fabricated DBRs with different periods. As the periods for the DBRs of
MgF$_2$-Nb$_2$O$_5$ bilayer films were with two, four, and six, the reflective ratios for a central wavelength of 450nm and calculated using Sheppard’s equation were 70.0%, 95.3%, and 99.3%. When the investigated overall transfer matrix was used to calculate the designed DBRs by the incorporations of n values and thicknesses, their reflective ratios were 68.0%, 94.3%, and 98.3% for central wavelengths of 436 (two periods), 456 (four periods), and 448nm (six periods). When the fabricated MgF$_2$-Nb$_2$O$_5$-based DBRs were used to measure their reflective ratios, they were 66.8% (for central wavelength of 436nm), 92.8% 456nm), and 95.3% (448nm). Regardless of whether the fabricated DBRs had different periods, all the deposited DBRs’ reflectivity had good uniformity, the deviations of the maximum reflective ratios between the measured results and the two simulation results could be neglectable.

When light strikes the surface of a film, part of light can be transmitted, part will be reflected, and the remaining part can be absorbed. The first law of thermodynamics defines that the sum of the transmitted, reflected, and absorbed radiation energies is equal to the energy of incident light. In other words, the relationships between absorptivity ($\alpha$), reflectivity ($\eta$), and transmissivity ($\tau$) are:

$$\alpha + \eta + \tau = 1$$  \hspace{1cm} (15)

In the past, Liu et al. used lossless Al$_2$O$_3$ and TiO$_2$ films (or k values close to zero in the visible light range, defined as $\alpha = 0$) to fabricate DBRs and found that the reflective ratios of the fabricated devices were higher than the simulation results using Sheppard’s approximate equation. For our DBRs fabricated using MgF$_2$-Nb$_2$O$_5$ bilayer films, maximum $\alpha$ plus maximum $\tau$ was smaller than 1. From the measurements of the transmission and reflection ratios, we can quantify that the total scattering and absorption losses at 450nm are $\alpha = 2.2\%$. These results prove that the higher k value of the Nb$_2$O$_5$ film meant the absorptivity ($\alpha$) was not equal to zero, so the reflective ratios of the MgF$_2$-Nb$_2$O$_5$ bilayer films were smaller than those obtained using Sheppard’s approximate equation.
Figure 11. Comparison of maximum reflective ratios using different simulation methods, and measurement results of fabricated DBRs with different periods.

4. Conclusions

When MgF$_2$-Nb$_2$O$_5$ bilayer films were used to fabricate DBRs with two, four, and six periods, the total thicknesses of the calculated MgF$_2$-Nb$_2$O$_5$ bilayer films were 259.8, 519.6, and 779.4nm, and the measured total thicknesses of the deposited MgF$_2$-Nb$_2$O$_5$ bilayer films were 242.8, 520.0, and 780.5nm. When we used two, four, and six as the period numbers to deposit the MgF$_2$-Nb$_2$O$_5$ bilayer films, the simulated maximum reflective ratios using Sheppard’s approximation equation were 70.0%, 95.3%, and 99.3%; using an overall transfer matrix, the values were 68.0%, 94.3%, and 98.3%, and the measured maximum reflective ratios were 66.8% (at 436nm), 92.8% (448nm), and 95.3% (456nm). The simulated bandwidth of the designed DBRs with MgF$_2$-Nb$_2$O$_5$ bilayer films at 450nm was 142nm. As the designed MgF$_2$-Nb$_2$O$_5$ bilayer films were two, four, and six periods, their simulated bandwidths with an overall transfer matrix were 103nm (390–493nm), 140nm (400–540nm), and 104nm (398–502nm), and the measured bandwidths for the deposited bilayer films were 88nm (385–497nm), 113nm (409–522nm), and 116nm (395–511nm). The results in this study prove that the investigated overall transfer matrix can be successfully used as a simulation tool to calculate the reflective spectra of fabricated MgF$_2$-Nb$_2$O$_5$ bilayer films.
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Figures

Figure 1

Structure of designed MgF2-Nb2O5 bilayer films with different periods.
Figure 2

XRD patterns of MgF$_2$ and Nb$_2$O$_5$ single-layer films and MgF$_2$-Nb$_2$O$_5$ bilayer film.
Figure 3

Measured (a) n values and (b) k values of glass substrate and single-layer MgF2 and Nb2O5 films; (c) and (d) K values of single-layer MgF2 and Nb2O5 films compared with Cauchy equation.
Figure 4

Surface morphologies of MgF2-Nb2O5 bilayer films with (a) two, (b) four, and (c) six periods.

Figure 5

Roughness analysis of MgF2-Nb2O5 bilayer films with (a) two, (b) four, and (c) six periods.
Figure 6

Cross-sectional observations of MgF2-Nb2O5 bilayer films with (a) two, (b) four, and (c) six periods.
Cross-sectional EDX mapping measurements of four-period MgF2-Nb2O5 bilayer films.
Figure 8

Measured reflectance rates of MgF2-Nb2O5 bilayer films as a function of period number.
Figure 9

Measured transmittance rate of MgF2-Nb2O5 bilayer films with different period numbers.
Figure 10

Simulated and measured reflectance spectra of DBRs designed using the MgF2-Nb2O5 bilayer films: (a) two, (b) four, and (c) six periods.
Figure 11

Comparison of maximum reflective ratios using different simulation methods, and measurement results of fabricated DBRs with different periods.