Analysis and experimental study of low-stepped mirror in light FTIRS*

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The structural parameters of low-stepped mirror which is the core component of a light Fourier transform infrared spectrometer (FTIRS) have important influence on the performance of the instrument. A simple method of multiple deposition is proposed to fabricate a low-stepped mirror with step height of 625 nm, for its high precision, high efficiency and miniaturization. So the film stress will be introduced in the process of coating, which can lead to step deformation. We build a bending model of the low-stepped mirror and analyze the influence of its deformation. The light intensity and spectrum are changed by the step deformation. Therefore, the film stress should be reduced in order to ensure the accuracy of the spectral information. Increasing the thickness of substrate is proposed in this paper to reduce the step deformation, besides adjusting the coating temperature and adding sub-layer. Our results reveal that the basic requirements of the system are satisfied when the thickness of the substrate is 5 mm in the appropriate coating environment. An FTIRS based on stepped mirrors and grid beam splitter is assembled and corresponding experiment is carried out to obtain the spectrum curve of acetonitrile.

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Spectrometry is a general physical-analysis approach for investigating light-matter interactions[1]. Compared with other types of spectrometers, Fourier transform infrared spectrometer (FTIRS) has a promising future in spectral analysis with advantages of multi-channel, high radiation flux, high precise wave number[2,3]. Hence, it has found increasing applications in many fields[4]. A space-modulated FTIRS based on a Michelson interferometer with two plane mirrors replaced by two stepped mirrors was proposed by Möller in 1992[5,6]. Subsequently, Centre National d'Etudes Spatiales (CNES) in French[7,8], Sardari reacherch in Turkey[9], including us[10] conducted detailed researches on them. The FTIRS mentioned above is static and can be miniaturized. It can not only be on-line monitored, but also realize real-time detection.

Two stepped mirrors are crucial components of the spectrometer, and their structural parameters significantly affect the instrument performance. So the fabrication and analysis of low-stepped mirror are of significance.

The MOEMS technology was chosen to fabricate the low-stepped mirror owing to its characteristic of millimeter-scale area and submicron height. We propose a method of multiple deposition to fabricate a low-stepped mirror consisting of 32 stages with a step height of 625 nm, which can precisely control the accuracy, consistency, and uniformity of the step height.

However, there is a problem of the step deformation caused by the film stress. It could cause a change of the optical path difference (OPD) which will affect the interferogram processing and the spectrum reconstruction. Therefore, we analyze the influence of step deformation on spectrum and propose the method of adding sub-layer, reducing temperature and increasing substrate thickness to reduce the film stress.

The experiments using different thicknesses of substrates are carried out, and the corresponding recovery spectra are obtained. A low-stepped mirror which meets the requirements of the system is selected. Our results will offer a reference for the fabrication of low-stepped mirror.

The simplified configuration of a light FTIRS is shown in Fig.1. The working principle of it is as follows. The light emitted from the light source propagated

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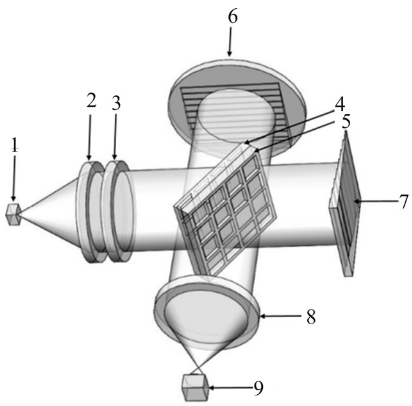
through collimating system and the sample cell, and then became parallel beam to reach the grid beam splitter system. The parallel beam incident on the grating-edge was absorbed by the absorption film. The beam incident into the grid was divided into two coherent beams, then they reached the tall-and low-stepped mirrors which were vertical to each other. They interfered when the beams returned to the grid beam splitter. The array of OPDs was imaged by the detector. Then the spectrum was obtained via Fourier transform of interferogram.



1. light source; 2. collimating system; 3. sample cell; 4. grid beam splitter; 5. compensating plate; 6. tall-stepped mirror; 7. low-stepped mirror; 8. expansion system; 9. infrared detector arrays

Fig.1 Simplified schematic of a light FTIRS

In the ideal case, the expression of the interference intensity is

$$I(m,n)=\int_0^\infty B(\nu)\exp[\mathrm{j}2\pi\nu\delta(m,n)]\mathrm{d}\nu , \tag{1}$$

where $I(m,n)$ is the interferogram intensity at the sampling space (m,n), m is the m-th step of low-stepped mirror, and n is the n-th step of the tall-stepped mirror. $B(\nu)$ is the power spectral density, ν is the spatial frequency of the optical signal, and $\delta(m,n)$ is the OPD of the sampling space (m,n).

The discrete Fourier transform (DFT) algorithm is used to obtain the spectral information. The expression of the spectral information is

$$B(\nu)=\sum_{m=1}^{32}\sum_{n=1}^{32}I(m,n)\exp[-\mathrm{j}2\pi\nu\delta(m,n)] . \tag{2}$$

The sketch map of low-stepped mirror is shown in Fig.2. The height, length and width of the step are represented by d, L and W, respectively. In order to assure the continuity of OPD, the height of a single step on a tall-stepped mirror is equal to the sum of all the step heights on low-stepped mirror, that is to say, the heights of the tall steps are expressed as Nd, N is the step number of low- and tall-stepped mirrors. So the sampling interval Δ is $2d$.

According to the Nyquist-Shannon sampling theorem, the sampling frequency must be greater than or equal to twice of the maximum frequency component of the signal being measured, that is to say, the sampling interval Δ is less than half of the minimum wavelength. For this system, the wavelength range is within 3—5 μm, and Δ is less than 1.5 μm. Then the height of low-stepped mirror is less than 0.75 μm. Therefore, the height d of low-stepped mirror is chosen as 0.625 μm. The spectral resolution is inversely proportional to the maximal OPD, which is expressed as $\delta_{\max}$. In our spectrometer, $\delta_{\max}=2N^2d$. To ensure that the spectral resolution is at least 8 cm^{-1}, $\delta_{\max}$ should be larger than 1 250 μm, i.e., $2N^2d\geq 1\,250$ μm. As a result, the value of sampling points N^2 should be greater than 1 000. In our system, we chose the number of the steps as 32, which can generate 1 024 sample points with different OPDs. Considering the effect of diffraction and the need for microminiaturization of spectrometers, the width W and the length L of low-stepped mirror are 1 mm and 32 mm, respectively.

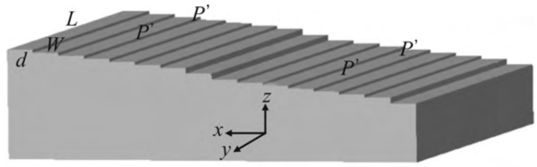


Fig. 2 Sketch map of low-stepped mirror

Because of the high accuracy and uniformity of the low-stepped mirror, the method of multiple deposition was used to manufacture the low-stepped mirror. The process is illustrated in Fig.3.

Firstly, a typical procedure of lithography is shown as follows: (1) Cleaning the substrate; (2) Spin-coating the photoresist to obtain a thick resist film whose thickness is greater than that of SiO_2 film; (3) Setting the sample on the hotplate and then pre-baking it; (4) Patterning the photoresist using a mask aligner; (5) Developing the sample using an AZ 400 K solution; (6) Rinsing the wafer with deionized water. This process is shown in Fig.3(a) and (b). The second step consisted of depositing an SiO_2 film with 10 μm thickness on the silicon wafers via electron beam evaporation, as shown in Fig.3(c). Next, acetone was used to remove the unexposed photoresist, after which we would obtain the two-step structure (Fig.3(d)). The low-stepped mirror with 32 steps could then be obtained by repeating the steps (a) to (d) for five times. It is noted that the width and height of film were halved gradually as the coating times increasing.

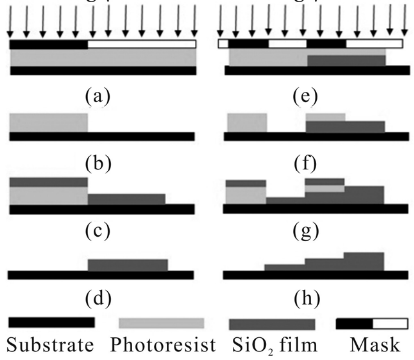


Fig.3 Schematic of the low-stepped mirror's fabrication process

According to the above steps, the low stepped-mirror was fabricated and tested. The roughness of the low-step mirror was measured by Nanosurf Core AFM, and the *RMS* was 1.72 nm. The test results show that the film thickness's accuracy and uniformity of each layer remained constant. However, the step was deformed by the film stress, during the manufacturing process of low-stepped mirror.

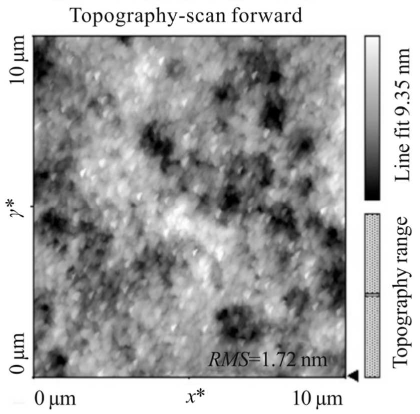


Fig.4 Roughness test result of low-stepped mirror

There were two kinds of stresses in the process of coating, namely, compressive stress and tensile stress. Stresses developed in films could lead to film delamination and blistering in the case of compression, while they could lead to cracking and peeling in the case of tension[11]. Experiments have demonstrated that low-stepped mirror was bearing compressive stress and the bending occurred along the direction of the sub-mirror, as shown in Fig.5.

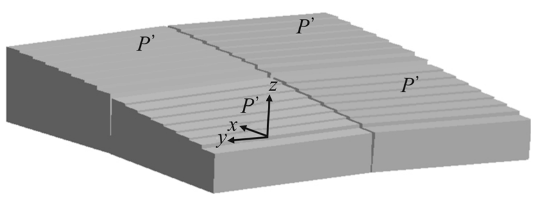


Fig.5 The low-stepped mirror model with deformation

In order to simplify the model, we assume that the deformation of the 32 steps is the same. Fig.6 shows one deformed step of low-stepped mirror, where $h(m,n)$ is the deviation-height of the step in the (m,n), t_s and t_f are the thicknesses of the substrate and film, respectively, and R is radius of curvature. As shown in Fig.6, the deviation-height is different with the position of step, and the maximal deviation-height is located in the middle of step. That is, the maximum additional OPD is located in the middle of the step. The relationship between $h_{\max}$ and R is

$$R^2=(R-h_{\max})^2+\frac{L^2}{4}\,. \tag{3}$$

When $R>>h_{\max}$, the above formula can be approximately written as

$$R=\frac{L^2}{8h_{\max}}\,. \tag{4}$$

From the Stoney formula, the film stress can be calculated by

$$\sigma_f=\left(\frac{E}{1-V}\right)\frac{t_s^{\,2}}{6Rt_f}\,, \tag{5}$$

where σ_f is the film stress, E is the Young's modulus of substrate, and V is the Poisson's ratio of substrate.

Replacing the expression of R into the above formula, the expression is

$$\sigma_f=\frac{4E}{3(1-V)}\frac{h_{\max}t_s^{\,2}}{t_f L^2}\,. \tag{6}$$

When the Young's modulus, the thicknesses of film and substrate, Poisson's ratio and length of step were fixed, the film stress could be obtained by measuring the maximal deviation-height. And the maximal deviation-height of step was decreased with the reducing film stress.

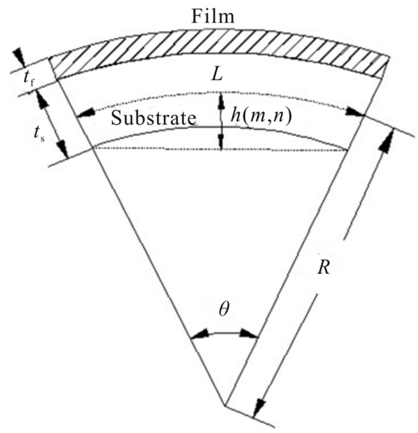


Fig.6 Bending model of deformed stepped mirror

Given that the additional OPD introduced by deformation of low-stepped mirror is $\Delta\delta(m,n)$, the expression of OPD in the spatial sampling (m,n) after being deformed is

$$\delta'(m,n)=\delta(m,n)+\Delta\delta(m,n)=$$
$$2d(Nn-m)-2h(m,n)\,. \tag{7}$$

Therefore, in the presence of step deformation, the light intensity obtained on the detector can be expressed as

$$I'(m,n)=\int_0^\infty B(\nu)\exp[\mathrm{j}2\pi\nu\delta'(m,n)]\mathrm{d}\nu=$$
$$\int_0^\infty B(\nu)\exp\{\mathrm{j}2\pi\nu[2d(Nn-m)-$$
$$2h(m,n)]\}\mathrm{d}\nu\,. \tag{8}$$

The real recovered spectrum with the deformation is then obtained via the DFT

$$B'(\nu)=\sum_{m=1}^{32}\sum_{n=1}^{32}I'(m,n)\exp[-\mathrm{j}2\pi\nu\delta(m,n)]\,. \tag{9}$$

From Eqs.(8) and (9), we can see that the light intensity and spectrum are changed by step deformation. When the deformation is large enough, the spectrum will be drifted.

Therefore, in order to ensure the accuracy of the spectral information, the stress of the film must be reduced.

A lot of researches have been done about the stress, including optimizing the material of the substrate, reducing the deposition temperature and rate[12,13]. We added a sub-layer of Al_2O_3 between the substrate and the film to reduce the mismatch. Reducing the coating temperature was also adopted to diminish the thermal stress. For the stepped mirror with 32 stages was obtained by multiple depositions, each coating condition had an effect on the film stress. That is to say, controlling the deposition parameters cannot completely solve the film stress above the low-stepped mirror. Therefore, the method of increasing the thickness of substrate, which can diminish the film stress according to Stoney Formula, was proposed in this paper.

According to the method mentioned above, we chose three groups of silicon chips as the base for the experiment. The thickness of each group of silicon chips was 1.5 mm, 3 mm, 5 mm, and the other experimental conditions were unchanged to eliminate the influence of other factors. Three groups of stepped structures with 32 stages were obtained by means of five times of lithography and coating.

The flatness of each group was measured by surface profilometer and the average value was obtained. The flatness results of thicknesses 1.5 mm, 3 mm and 5 mm are shown in Fig.7. It can be seen that the closer to the step center, the greater the step deformation. The details are shown in Tab.1.

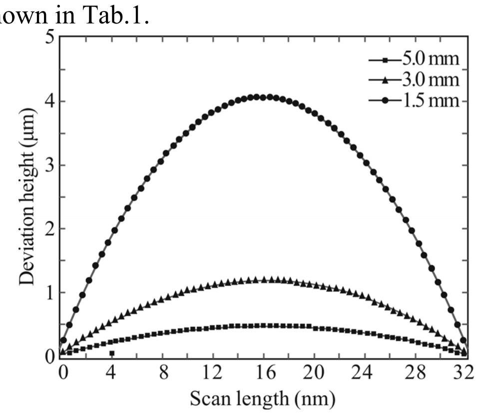


Fig.7 Flatness test results of the low-stepped mirror with different substrate thicknesses

Tab.1 Results of flatness test

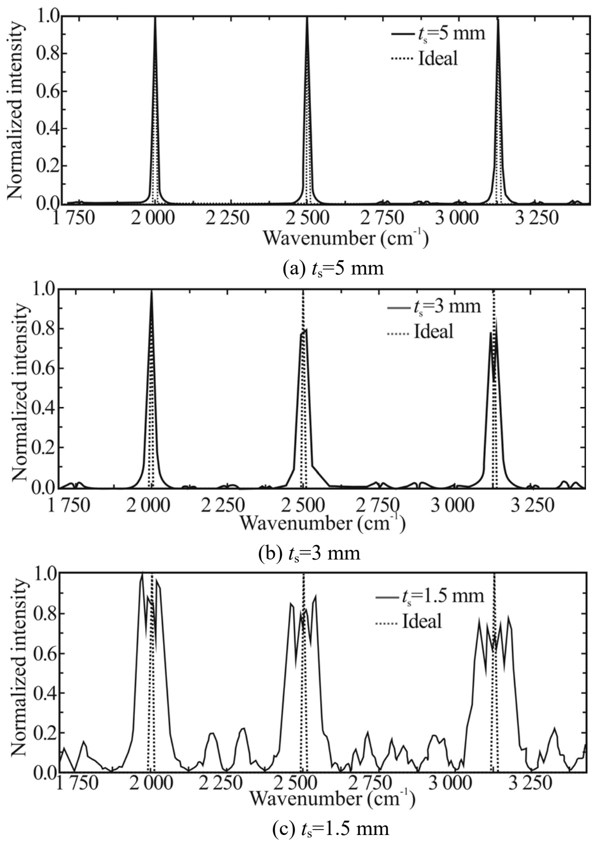
Parameter	Value		
t_s/mm	5	3	1.5
h_{max}/μm	0.49	1.188	4.055

The light FTIRS was simulated in ASAP software using the above data and the monochromatic light was taken as an example. The interferogram obtained from the detector was expanded into one dimensional OPD sequences in order. And Fourier transform was performed. As shown in Fig.7, the recovery spectra at different substrate thicknesses when the wavelengths are 3.2 μm, 4 μm and 5 μm were obtained.

When the substrate thickness is 5 mm, there is no deviation in peak position at all wavelengths, in which condition the spectrum will be reconstructed well. When the substrate thickness is 3 mm, the width of spectrum is broadened and the spectral curves of 3.2 μm and 4 μm show double-peak, while the peak position of 5 μm has no deviation. When the substrate thickness is 1.5 mm, the spectrum curve in the whole wavelength band is distorted. With the decrease of substrate thickness, the peak position of spectrum is shifted. For the same substrate thickness, the degeneration of spectrum curve of short wavelength is larger than that of long wavelength.

The full width at half maximum (*FWHM*) can describe the spectral resolution of SMFTIRS. Fig.8 describes the *FWHM* for different maximum deviation-heights at wavelengths of 3.2 μm, 4 μm and 5 μm. As can be seen from Fig.8, with the increase of h_{max}, the spectral curve is broadened, which means that the spectral resolution of the instrument is decreased.

From the above results, it can be seen that for the low-stepped mirror, the step's deformation can cause the decrease in the instrument's spectral resolution and the spectral line drift. Fig.8 and Fig.9 show that when substrate thickness is 5 mm, the peak position of spectrum does not change and a high spectral resolution is obtained. In our system, the detector's response wavelength is from 3.7 μm to 4.8 μm, to which the analysis is also applicable.



(a) t_s=5 mm

(b) t_s=3 mm

(c) t_s=1.5 mm

Fig.8 Spectral curves at different t_s values when the wavelength is 3.2 μm, 4 μm and 5 μm

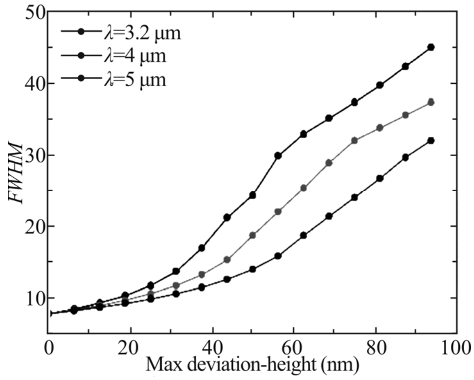


Fig.9 Variation of *FWHM* with maximum deviation-height when the wavelength is 3.2, 4 and 5 μm

The low-stepped mirror with the 5 mm substrate thickness was deposited on golden film as the reflective coating as shown in Fig.10. The details of step height are listed in Tab.2. The maximum, minimum, average and standard deviation are included.

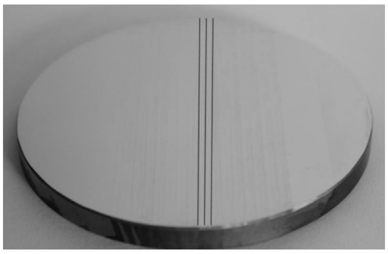


Fig.10 The low-stepped mirror coated with gold reflector

Tab.2 The detail of step height

Step height	d_{max}	d_{min}	d_{aver}	Standard deviation σ
Values (nm)	649	618	626.9	7.4

The setup was built and experiments have been done to obtain the spectrum of liquid acetonitrile (CH_3CN). The prototype of the light FTIRS is shown in Fig.11. As a result, the interferogram recorded by the detector array was obtained. The absorption interferogram of the sample can be obtained by using the difference between the interferogram without the sample and the interferogram with the sample. After the interferogram was processed[14], the absorption spectrum of the sample can be obtained in Fig.12 by performing Fourier transform.

Comparing the spectrum of Fig.12 with the standard spectrum of liquid CH_3CN supplied by National Institute of national standards and technology (NIST)[15], it is found that the positions of the main peaks coincide with each other at 2 258 cm^{-1}. This indicates that the low-stepped mirror we fabricated can be applied to the light FTIRS system. But the spectrum we provided is broadened, which is caused by insufficient spectral resolution. Therefore, it is necessary to increase the step number of low stepped-mirrors to improve spectral resolution, which is a new challenge for MEMS technology.

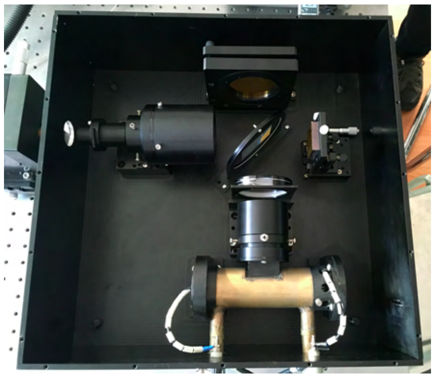


Fig.11 Simplified configuration of the light FTIRS

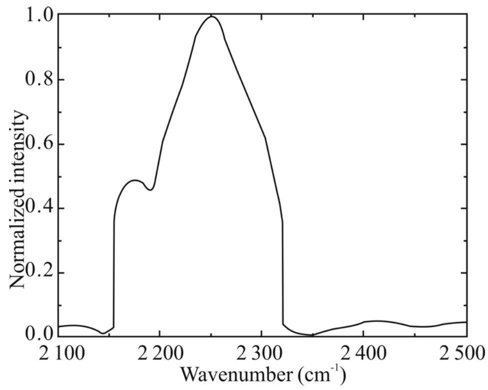


Fig.12 Spectral curve of sample CH_3CN

Stress in films is the primary reason for the step deformation which could affect the OPD in a stationary light FTIRS. Our study has shown that the step deformation can influence the spectrum. In order to reduce the compressive stress, the method of adding sub-layers and adjusting the temperature of deposition is adopted. In addition, increasing the thickness of the substrate is effective in improving its shape by decreasing the deformation. Results of our experiments have revealed that the peak position of the spectrum does not drift when thickness of a substrate is 5 mm. The low-stepped mirror is assembled in the FTIRS, and the experiment is carried out. The spectral line of a CH_3CN is obtained with an accurate peak position. Our study will offer a reference for the fabrication of low-stepped mirrors.

References

[1] Jacob Reimers, Aaron Bauer, Kevin P Thompson and Jannick P Rolland, Light: Science & Applications **6**, e17026 (2017).

[2] Cao Dian-sheng, Shi Zhen-hua and Lin Guan-yu, Optics and Precision Engineering **25**, 6 (2017). (in Chinese)

[3] Sun Li-wei, Ye Xin and Fang Wei, Optoelectronics Letters **13**, 405 (2017).

[4] M. Yan, P.L. Luo, K. Iwakuni, G. Millot, T.W. Hänsch and N. Picque, Light Science & Applications **6**, e17076 (2017).

[5] Möller K. D., SPIE **1992**, 130 (1993).

[6] Möller K. D., Appl. Opt. **34**, 9 (1995).

[7] Brachet F., Hébert P.J. and Cansot E., SPIE **7100**, 710019 (2008).

[8] Lacan A., Bréon F.M., Rosak A., Brachet F., Roucayrol L., Etcheto P., Casteras C. and Salaün Y., Opt. Express **18**, 8 (2010).

[9] Behzad Sardari, Federico Davoli and Meriç Özcan, Review of Scientific Instruments **87**, 97 (2016).

[10] Liang Jing-jiu, Liang Zhong-zhu, Lv Jin-guang, Qin Yu-xin, Tian Chao and Wang Wei-biao, Chinese Optics **8**, 2 (2015). (in Chinese)

[11] Li Dong-ling, Feng Xiao-fei, Wen Zhi-yu, Shang Zheng-guo and She Yin, Optoelectronics Letters **12**, 285 (2016).

[12] Dong Kai-chen, Lou Shuai, Yao Jie, Wu Jun-qiao and You Zheng, Optics and Precision Engineering **26**, 1 (2018). (in Chinese)

[13] Y. Q. Ji, Y.G. Jiang, H. S. Liu, L. H. Wang, D. D. Liu and C. H. Jiang, Thin Solid Films **545**, 10 (2013).

[14] Gao J. H., Liang Z. Z., Liang J. Q., Wang W. B, Lv J. G. and Qin Y. X., Applied Spectroscopy **71**, 6 (2017).

[15] https://webbook.nist.gov/cgi/cbook.cgi?ID=C75058&Units=SI&Type=IR-SPEC&Index=2#IR-SP