Acta Mechanica Sinica (2012) 28(5):1382–1388
DOI 10.1007/s10409-012-0146-z

RESEARCH PAPER

Effect of ion-beam assisted deposition on the film stresses of TiO_2 and SiO_2 and stress control

Yu-Qiong Li · Hua-Qing Wang · Wu-Yu Wang · Zhi-Nong Yu · He-Shan Liu · Gang Jin

Received: 11 November 2011 / Revised: 31 March 2012 / Accepted: 25 June 2012

Abstract Based on Hartmann–Shack sensor technique, an online thin film stress measuring system was introduced to measure the film stresses of TiO_2 and SiO_2, and comparison was made between the film stresses prepared respectively by the conventional process and the ion-beam assisted deposition. The effect of ion-beam assisted deposition on the film stresses of TiO_2 and SiO_2 was investigated in details, and the stress control methodologies using on-line adjustment and film doping were put forward. The results show that the film stress value of TiO_2 prepared by ion-beam assisted deposition is 40 MPa lower than that prepared by conventional process, and the stress of TiO_2 film changes gradually from tensile stress into compressive stress with increasing ion energy; while the film stress of SiO_2 is a tensile stress under ion-beam assisted deposition because of the ion-beam sputtering effect, and the film refractive index decreases with increasing ion energy. A dynamic film stress control can be achieved through in-situ adjustment of the processing parameters based on the online film stress measuring technique, and the intrinsic stress of film can be effectively changed through film doping.

Y.-Q. Li · H.-S. Liu · G. Jin (✉)
National Microgravity Laboratory (NML),
Institute of Mechanics, Chinese Academy of Sciences,
100190 Beijing, China
e-mail: gajin@imech.ac.cn

H.-Q. Wang
Optical Thin Film Center,
Daheng New Epoch Technology Inc., 100070 Beijing, China

W.-Y. Wang
Beijing General Research Institute for Nonferrous Metals,
Division of Mineral Research and Material Energy,
100088 Beijing, China

Y.-Q. Li · Z.-N. Yu
School of Optoelectronics,
Beijing Institute of Technology, 100081 Beijing, China

Keywords Film stress · Stress controlling · Ion-beam assisted deposition · Hartmann–Shack sensor

1 Introduction

It is common for film stresses to occur in films during their preparing processes, and almost all films have some kinds of stresses. The factors affecting the film stresses are diversified, including film material, substrate, deposition process and environmental parameters [1–4], while the deposition process is one of the most important factors to induce the film stresses, so the effect of deposition process on film stresses and stress control method is an important subject. Film stresses can result in curved optical films, and make the reflected light distorted [5]. Even worse, the film stresses can cause the films to be cracked and peeled off, making the films completely invalid.

Ion-beam assisted deposition (IBAD) has become a preferred method for producing high quality optical films. There are many reports about IBAD influencing the characteristics of oxide films and optical films [6, 7], not only increasing the density and eliminating the columnar crystal structure of films, but also improving the stability and uniformity of the films' optical constants and improving the films' stoichiometric ratio and so on. However, there are few reports about the effect of IBAD process on film stresses, and corresponding investigations on film stresses control are also rare.

Now the main film stresses measuring methods include substrate deformation method [8], diffraction method [9], spectrometry and film vibration method [10]. The substrate deformation method is widely adopted in the film stress measuring field for its simplicity and non-destructiveness. At present, the measuring method of substrate deformation are mostly based on off-line measurement. For example, Shao et al. [11] developed a testing device of film stress using the reflecting principle of light; Chen et al. [12] reported that a whole cascade film stress measurement device of moiré fringe had been developed; Bicker et al. [13] have proposed a in-situ film stress measurement method using double reflecting lights, and the sensitivity of which is only 4.4 MPa; Lin et al. [14] developed a film stress sensor of dual beam array, mainly using the 2D Dammann grating and Fresnel wave band technologies, and the measuring sensitivity of which is 2.5 MPa. Since the real-time film stress measuring devices need to be installed on the film preparing equipments, the sensitivity of these methods will be affected by the vibrations of the equipments, limiting the application of these methods in many aspects. In this paper, an online film stresses measuring system based on Hartmann–Shack (H–S) sensor technique is adopted. The equipment is non-sensitive to the vibrations of the device during the film preparing process because the measuring principle is calculating the relative deformation of each very small area of samples. It makes the precise measurement and real-time measurement of film stresses feasible.

In this paper, the film stresses of TiO_2 and SiO_2 were analyzed and compared respectively under the conventional process and the IBAD conditions based on a real-time film stresses measuring system of H–S sensor, and the effect of ion energies on the film stresses was studied. Also, stress control methodologies using in-situ adjustment and film doping were put forward. The film stresses measurement is a complicated work since the effect of IBAD on film stresses is complex, and the exploratory work presented in this paper is only for reference.

2 Experiment

The ZZSX-800ZA automatic vacuum coating machine and in-situ film stress measurement system based on H–S were adopted in this experiment, the structure of which were shown in Figs. 1 and 2, respectively. The H–S film stress instrument was installed on the top of the coating machine, and the computer connected with the CMOS camera was responsible for processing single data collected by the CMOS camera. The system is consisted of three parts: the self-collimator imaging system, the H–S sensor and the image acquisition and processing unit. The light emitted from the light source passes through the prism, then normally incidents on the sample's surface after passing through the objective lens. The parallel light is reflected by the sample's surface, then changes into parallel light after passing through relay lens. Finally, imaging on the surface of the photodetector after passing through the H–S lens array. The basic principle of the instrument is as follows: the sample's surface is divided into several small areas by the H–S lens array to get the small areas' slope caused by the film stresses, and through measuring the relative position change of each small region's image, the whole area's deformation is obtained. When the film thickness is far less than that of the substrate, we can get the film stresses from the following stress formula proposed by Manning et al. [15, 16]

$$\sigma_f = \frac{4E_s t_s \delta}{3(1-\eta_s)D_S^2 t_f},$$

where E_s, t_s, δ, D_s, η_s and t_f are the substrate elastic modulus, the substrate thickness, the deformation slope of the substrate, the diameter of the substrate, the Poisson's ratio and the film thickness, respectively.

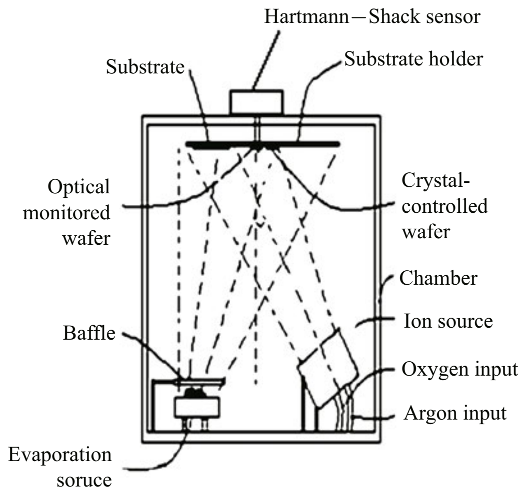

Fig. 1 The schematic scheme of ZZSX-800ZA automatic vacuum coating machine

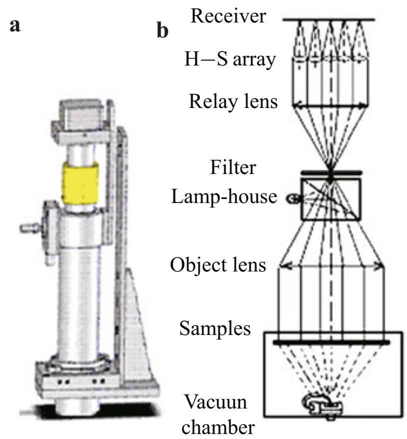

Fig. 2 THe physical scheme and structural drawing of no-line film stress measuring instrument based on Hartmann–Shack sensor: **a** Physical scheme; **b** Structural drawing

As to the measurement precisions of this system, the measurement precision of substrate deformation slope is 15.6 nm, and the measurement precision of film stress is 3.3 MPa (BK7, 0.7 mm thickness); the measurement stability of light spot centroid is 0.52 μm (1/10 CMOS pixels) for 2 h, and the measurement frequency is 6 Hz. Taking the light spot of the sample center as the referring point, we measure the variations of other spots relative to the referring point. This system can avoid measuring error when the samples are vibrating through adaptation.

The Kaufman ion source made by Space Science and Applied Research of Chinese Academy of Science is adopted in this experiment, the diameter of which is 12 cm, and the ion beam energy is 100–600 eV. BK7 glass is used as the substrate, the size of which is Φ 40 mm × 0.7 mm. The elastic modulus of the substrate is $E_s = 75$ GPa, and the Poisson's ratio $\eta_s = 0.21$. The substrates are cleaned with commercial detergent and de-ionized water before loading, then the substrates are pre-cleaned using Ar^+ ion-beam bombardment for 5 min to further reduce impurities on the substrate surfaces prior to deposition. The Ar^+ ion-beam parameters for cleaning are: ion energy of 350 eV, ion beam current of 100 mA, argon flux of 5 mL/min. A 15 nm silver film was coated on the substrate surface which was taken as the stress testing surface, and then put in the atmosphere 72 h for aging treatment. The refractive indexes of films are measured by post-deposition ellipsometry measurements (VASE, J.A. Woollam Co., Inc.).

3 Results and discussions

3.1 The effect of IBAD on the film stresses of SiO_2 and TiO_2

3.1.1 Comparisons between film stresses of SiO_2 and TiO_2 films prepared respectively by IBAD and traditional process

TiO_2 and SiO_2 films are the research objects, with the BK7 glass as the substrates. The preparation parameters are shown in Table 1. TiO_2 and SiO_2 in the table mean that the TiO_2 and SiO_2 films prepared by traditional process, and TiO_2/IBAD and SiO_2/IBAD in the table mean that the TiO_2 and SiO_2 films prepared by IBAD. The film stresses are online monitored during the preparation.

Table 1 Preparation parameters of TiO_2 and SiO_2 films

Films	Ion source		Gas flow		Evaporation rate/($nm \cdot s^{-1}$)	Vacuum/(mPa)	Substrate temperature/°C
	Accelerating voltage/V	Beam current/mA	O_2/($mL \cdot min^{-1}$)	Ar/($mL \cdot min^{-1}$)			
TiO_2	0	0	30	0	0.3	15	250
TiO_2/IBAD	330	100	25	5	0.3	20	250
SiO_2	0	0	0	0	6	0.6	250
SiO_2/IBAD	330	100	8	5	0.6	12	250

Usually, the types of film stresses are defined by the substrate deformation compared to that of the film. When the substrate shrinks towards the film, this kind of film stress is called tensile stress (the stress value is "+"); while the substrate shrinks opposite to the film, this kind of film stress is called compressive stress (the stress value is "–").

From Fig. 3 we can see that the stress of the TiO_2 film prepared by the traditional process is a compressive stress in the beginning, then it changes into tensile stress when the film is 30 nm thick, and rises slowly with increasing thickness. The stress reaches the maximum value of 150 MPa when the thickness is 150–175 nm, then decreases slowly. When the thickness is 300 nm, the stress value is 124 MPa. The stress of TiO_2 film prepared by IBAD is also a compressive stress in the beginning, however, the compressive stress changes into tensile stress when the film thickness is only 18 nm. While the thickness is greater than 30 nm, the rising rate becomes slow and stable, and the stress value is only 84.8 MPa when the thickness is 300 nm.

Meanwhile, the following two conclusions can be drawn from Fig. 3 for SiO_2 films: (1) The film stress of SiO_2 prepared by IBAD declines more sharply compared to that prepared by traditional process; (2) The film stress of SiO_2 can be changed from compressive stress into tensile stress under an appropriate condition.

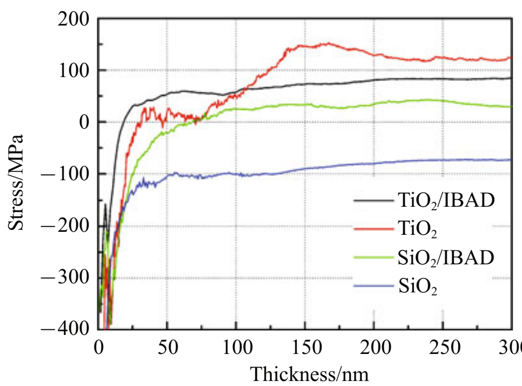

Fig. 3 Relationship between thickness and film stresses of TiO_2 and SiO_2 under two preparation conditions

The film stress of TiO_2 is tensile stress under both the preparation conditions, and the stress value of TiO_2 film prepared by IBAD is 40 MPa lower than that of TiO_2 film prepared by traditional process; but the film stresses of SiO_2 prepared by the two preparation conditions are greatly different. The film stress of SiO_2 prepared by traditional process is compressive stress, while that of SiO_2 film prepared by IBAD is tensile stress. This conclusion is different from the common conception that the structure of films prepared by IBAD is denser than that of the films prepared by traditional process. Preliminary hypothesis results from the sputtering effect of ion-beam, since the structure of SiO_2 is denser than that of TiO_2 [17], and the evaporating rate of SiO_2 is larger than that of TiO_2. So it is easier for sputtering effect of ion-beam to occur in SiO_2 films, and the sputtering effect is more obvious. The effect of IBAD on different films is distinctly different, depending on the properties and the preparation parameters of the film itself.

3.1.2 The effect of ion source parameters on the film stresses

Using different ion energies, and the ion energies are characterized with the accelerating voltage of ion source in this experiment. The preparation parameters of the samples are the same except for the accelerating voltage of ion-beam, and the preparation temperature is room temperature, as shown in Table 1. The accelerating voltages of ion source used in the experiment are 220 V, 330 V, 450 V and 550 V, respectively. The film stresses are on-line monitored during preparation.

TiO_2 films have complicated lattice structure and crystal structure (e.g., anatase, rutile, brookite), varying with the substrate temperature and particle energy. TiO_2 films can easily crystallize when the films are forming (anatase phase), so it will result in instable refractive index of TiO_2 films (the filling density of films changes with the change of crystallization phase).

The relationship between film stresses of TiO_2 films and ion source energies is shown in Fig. 4. It shows that TiO_2 film stresses gradually change from tensile stress into compressive stress with increasing ion energy. The type of stress changes when the ion energy is 450 eV. When the ion energies are 200 eV, 330 eV, 450 eV and 550 eV, the corresponding refractive index of TiO_2 films are 2.441, 2.563, 2.561 and 2.452, respectively (λ = 600 nm). Considering the film stress and refractive index comprehensively, it can be concluded that the stress type of TiO_2 film changes and the refractive index reaches its maximum value when the ion energy is about 450 eV. Therefore, it can be inferred that the crystal structure of TiO_2 films changes from polycrystalline state into amorphous state. Meanwhile, it also shows that the effects of ion implantation and sputtering on TiO_2 films increase with increasing ion energies, which results in a gradual decline of the refractive index of TiO_2 films.

The relationship between film stresses of SiO_2 films and ion energies is shown in Fig. 5. It shows that the stress type of SiO_2 films changes gradually from compressive stress into tensile stress with increasing ion energy. The stress value gradually declines when the ion energy exceeds 330 eV, and the stress tends to be stable when the ion energy is above 450 eV.

IBAD can make the stress of SiO_2 change, and make the film refractive index decrease with increasing ion energy. It is mainly due to the sputtering effect of ions, which makes the compacted structure of films become loose. When the ion energy reached a given value (e.g., 330 eV), the effects of ion implantation appeared immediately, and made the residual stress value decrease.

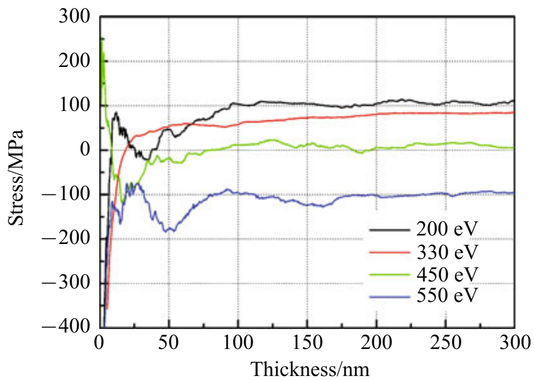

Fig. 4 Relationship between thickness and film stresses of TiO_2 films prepared under different ion energies

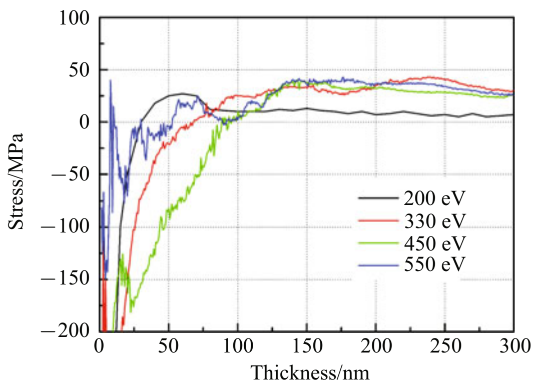

Fig. 5 Relationship between thickness and film stresses of SiO_2 films prepared under different ion energies

3.2 Film stresses control

3.2.1 In-situ adjustment

The main principle of film stresses control techniques through on-line adjusting preparation parameters is based on the in-situ film stress measuring system, which is developed by our laboratory. According to in-situ measurement of substrate deformation or film stresses, we can adjust the preparation parameters of the subsequent films to control the substrate deformation and mean the stress of the total structure in time. Firstly, find out the relations between the film stress and preparation parameters (e.g., deposition rate, chamber pressure, ion source parameters, etc.). Secondly, according to the actual measurements of substrate deformation or stress value in the film preparing process, we adjust the preparation parameters of films in real-time, and make the development of substrate deformation or stress value to the desired direction. Then, we should take care of the rationality of the adjusted parameters, and consider the effect of the adjustment of processing parameters on the optical properties of films, because the changes should be limited to a suitable range.

From Sect. 3.1 we can know that the film stresses of TiO_2 and SiO_2 vary greatly with varying ion energies. In a previous work, we found that the film stress would transform rapidly from tensile stress into compressive stress with increasing vacuum chamber pressure when the pressure was lower than 13 mPa; while the film stress would transform slowly from compressive stress into tensile stress with increasing vacuum chamber pressure when the pressure was higher than 13 mPa [17]. Therefore, by regulating the ion energy and vacuum chamber pressure during the preparing process we can adjust the film stress of device structure.

The film stress control process for the preparation of high reflective optical films can be described as follows. The structure system is designed as: Glass/5(HL)/Air, H : TiO_2, d = 51.6 nm; L : SiO_2, d = 85.7 nm. Table 2 shows the preparation parameters of each film. Figure 6 shows the relationship between the substrate deformation and the film thickness. From the chart, we can see that after 1#TiO_2 and 2#SiO_2 has been prepared, the total film stress is tensile stress, so we should increase the accelerating voltage and decrease the vacuum pressure to increase the density of film. When 4#SiO_2 has been prepared, the film stress is still tensile stress, so we should further increase the accelerating voltage for subsequent films. When 5#TiO_2 has been finished, the film stress becomes compressive stress, we should decrease the accelerating voltage and increase the vacuum pressure to make the following films loose. When 7#TiO_2 has been finished, the deformation of substrate is close to zero. The preparation parameters for subsequent films can be adjusted in a reasonable range according to the above principle to make the deformation of substrate close to zero. As seen from the chart, the cumulative deformation of the substrate is less than 1 nm.

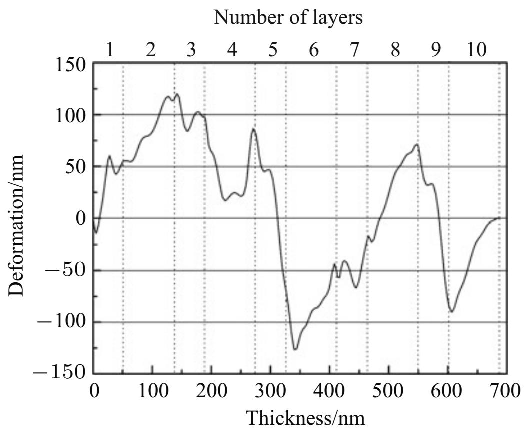

Fig. 6 Relationship between thickness and deformation of the substrate

Table 2 Processing parameters of films adjusted on-line

Serial number	Films	Thickness/nm	Ion source		Vacuum/mPa
			Accelerating voltage/V	Beam current/mA	
1	TiO_2	51.6	330	100	19
2	SiO_2	85.7	330	100	13
3	TiO_2	51.6	450	100	17
4	SiO_2	85.7	450	100	11
5	TiO_2	51.6	550	100	17
6	SiO_2	85.7	200	100	13
7	TiO_2	51.6	200	100	19
8	SiO_2	85.7	330	100	11
9	TiO_2	51.6	550	100	19
10	SiO_2	85.7	300	100	11

3.2.2 Doping effect

The intrinsic stress of film will be changed greatly by doping with certain proportion of impurities. There are two theories to explain this phenomenon. One is that the intrinsic stress of film is caused by the electron density difference between the film and the substrate surface, which results from the continuity of atom surface electron density in the interface. This mode is known as the electron density contrast of atomic surface mode [18, 19]. So we can reduce the stress of the film by injecting ions according to this theory [20]. The other is that the intrinsic stress of film is influenced by the crystal lattice structure of the film [21], so doping certain proportion of impurities will optimize the crystal lattice structure and reduce the intrinsic stress of the film [22–25].

In this experiment, zirconia oxide (ZrO_2) and titanium oxide (TiO_2) are adopted to carry out experiments. The mixed oxide consists of ZrO_2 and TiO_2 with a mass ratio of 9:1, which is provided by the Beijing General Research Institute of Nonferrous Metals. The preparation parameters of ZrO_2, TiO_2 and ZrO_2+TiO_2 mixed films are shown in Table 3, and the relationship between stress and thickness of the three films is shown in Fig. 7.

From Fig. 7 we can see that the stress of the ZrO_2+TiO_2 mixed film is 104 MPa when it is 200 nm, decreased by

71 MPa compared to the pure ZrO_2 film. The reasons are as follows: While TiO_2 is doped into ZrO_2 to prepare a film, the atoms of TiO_2 filling into the holes of ZrO_2 crystal structure to form a mosaic structure, which plays an important role in resisting the stress deformation for the host material of ZrO_2, so TiO_2 optimize the crystal lattice structure and reduce the intrinsic stress of ZrO_2 film [26, 27]. By measuring the optical parameters of films, the optical properties (e.g., transmittance and refractive index) of ZrO_2+TiO_2 mixed film change little compared to the pure ZrO_2 film.

Table 3 Preparation parameters of ZrO_2, TiO_2 and ZrO_2+TiO_2 films

Films	Ion source		Gas flow		Evaporation	Vacuum/mPa
	Accelerating voltage/V	Beam current/mA	$O_2/(mL\cdot min^{-1})$	$Ar/(mL\cdot min^{-1})$	rate/$(nm\cdot s^{-1})$	
ZrO_2	330	100	8	5	0.3	15
TiO_2	330	100	25	5	0.3	20
ZrO_2+TiO_2	330	100	8	5	0.3	15

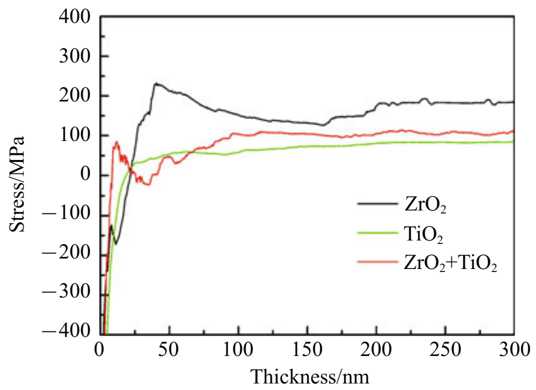

Fig. 7 Comparison between the film stresses of ZrO_2, TiO_2 and ZrO_2+TiO_2 mixed films

4 Conclusions

The film stress investigation is important and promising, and some exploratory work about the effect of IBAD on the film stress and film stress control methodologies have been studied. The film stresses of TiO_2 and SiO_2 films prepared by IBAD are different from those of the films prepared by traditional process. The film stress of TiO_2 film is tensile stress under both the preparing conditions, but the stress value of TiO_2 film prepared by IBAD is 40 MPa lower than that of which prepared by traditional process. The film stress of SiO_2 film prepared by traditional process is compressive stress, while that of which prepared by IBAD is tensile stress. With increasing ion energy, the structure of the TiO_2 film becomes more compact, and the stress changes from tensile stress into compressive stress, and the refractive index also increases. However, the effect of IBAD on the SiO_2 film is opposite.

Meanwhile, according to in-situ measurement of substrate deformation or film stress, adjusting the preparation parameters of each film can control the substrate deformation and mean the stress of the total structure in time. By doping with certain proportion of impurities, the intrinsic stress of film will be changed greatly, and the film stress will be adjusted effectively.

References

1 Gu, P. F.: Thin Films Technology. Zhejiang University Press, Hangzhou (1990) (in Chinese)
2 Alfons, Z., Rainer, G., Matl, K.: Plasma ion assisted deposition: Investigation of film stress. Proc. SPIE **2776**, 207–211 (1996)
3 Petar, V.: Mechanical stresses in oxide thin films. Vacuum **43**, 727–729 (1992)
4 Shao, S. Y., Fan, Z. X.: Evolutions of residual stress and microstructure in ZrO_2 thin films deposited at different temperature and rates. Thin Solid Films **445**, 59–62 (2003)
5 Anthnoy, E. E.: Stress developed in optical film coatings. Applied Optics **5**, 51–61 (1966)
6 Michael, L. F.: Application of ion-assisted deposition using a grid-less end-Hall ion source for volume manufacturing of thin film optical filters. Proc. SPIE **2253**, 374–393 (1994)
7 Hansjoerg, S. N., N. K., Uwe, B. S., et al.: IAD of oxide coatings at low temperature : A comparison of process based on different ion sources. Proc. SPIE **3133**, 205–213 (1997)
8 Stoney, G. G.: The tension of thin metallic film deposited by electrolysis. Proc. of the Royal Society of London **82**, 172–180 (1909)
9 Malhotra, S. G., Rek, Z. U., Yalisove, S. M., et al.: Analysis of thin film stress measurement techniques. Thin Solid Film **301**, 45–54 (1997)
10 Wen, Z., Cao, X. J.: Current status of research on measurement of the stress in thin film. Machinery Manufacturing Engineer **4** 127–129 (2005) (in Chinese)
11 Shen, Y. M., Zhu, M. P., Shao, S. Y.: A high precision thin film stress on-line measurement structure and measuring method. **36**, 412–415 (2007) (in Chinese)
12 Chen, K. S., Chen, T. Y. F., Chuang, C. C., et al.: Full-field wafer lever thin film stress measurement by phase-stepping shadow moire. IEEE Transactions on Components and Packaging Technologies **27**, 594–603 (2004)
13 Bicker, M., Von Hülsen, U., Laudahn, U., et al.: Optical deflection stress measurements in thin films. Review of Scientific Instruments **69**, 460–462 (1998)
14 Lin, X. C., Guo, Y. Y., Hu, Z. M., et al.: A kind of thin-film stress sensor based on beam-focusing multi-beam array. Chin. J. Semi. **25**, 1491–1494 (2004) (in Chinese)
15 Manning, A. S., Fuchs, S.: Finite element analysis of thermal stresses in high-power substrates for hybrid circuits. Materials

& Design **18**, 61–72 (1997)
16 Callioğlu, H., Bektag, N. B., Sayer, M.: Stress analysis of functionally graded rotating discs: Analytical and numerical solutions. Acta Mechanica Sinica **27**, 950–955 (2011)
17 Li, Y. Q., Yu, Z. N., Wang, H. Q., et al.: Effects of substrate materials and deposition parameters on film stress. Acta Optica Sinica **30**, 602–608 (2010) (in Chinese)
18 Cheng, K. J., Cheng, S. Y., et al.: Analysis and computation of the internal stress in thin films. Progress in Natural Science **8**, 679–689 (1998)
19 Cheng, S. Y., Cheng, K. J.: Computation on heat of formation and EOS of alloy by are fined TFI model. Acta Phys. Sin. (Oversea Edition) **2**, 439 (1993)
20 Wang, R. N., Liu, J. F.: The study of the effect of C^+ ion injection on the film stress of $CoSi_2$. Progress in Natural Science **12**, 1296–1300 (2002) (in Chinese)
21 Cheng, Y. M., Tang, J. F., Gu, P. F.: Measurement and analysis of properties of films doped with oxides. Acta Optica Sinica **6**, 70–75 (1986) (in Chinese)
22 Pulker, H. K.: Characterization of optical thin films. Thin Solid Films **18**, 1969–1977 (1979)
23 Zhang, G. B., Hao, Y. L., Tian, D. Y., et al.: Study on characteristics of poly-silicon film stresses. Chinese Journal of Semiconductors **20**, 463–467 (1999) (in Chinese)
24 Li, R. B.: Study of the stress in doped CVD diamond films. Acta Physica Sinica **56**, 3429–3434 (2007) (in Chinese)
25 Ma, B. X., Yao, N., Jia, Y., et al.: Influence of structure on adhesion of grains in CVD diamond films. Acta Physica Sinica **54**, 2854–2858 (2005) (in Chinese)
26 Luo, Y. H., Liu, C. H., Wang, W. Y.: Titanium Compounds. Metallurgical Industry Press, Beijing (2011) (in Chinese)
27 Ceniga, L.: Thermal stresses in two- and three- component anisotropic materials. Acta Mechanica Sinica **26**, 695–709, 2010