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Stress and Microstructure Study of W/Si X-ray Multilayers with Different Structural Parameters

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X-ray Timing and Polarimetry (XTP) telescope is proposed by using nested focusing optics with a large effective area for studies in the field of high energy physics (energy region of 1–30 keV). High-reflectance and low-stress W/Si multilayers are required for this telescope to improve the spectral response and maintain the figure quality of the mirrors simultaneously. In this paper, we focused on the study of stress and layer structure of W/Si periodic multilayers at different d -spacings (thickness of period), thickness ratios, and bilayer numbers. The results show that the stress of the multilayer increased from -73.3 to -465.5 MPa with increase in the d -spacing from 2.7 to 5.5 nm, while the change in the average interface width was negligible, from 0.31 to 0.36 nm. For the multilayers prepared with different thickness ratios of W, from 0.3 to 0.67 ($d = 3.7$ nm), the lowest stress appeared at the ratio of ~ 0.46 , and the average interface width was unchanged. The number of bilayers ($N = 80$ –160) had negligible effects on both the layer structure and the stress, and the multilayers exhibited a very smooth surface morphology with a root-mean-square roughness of 0.19 nm. To further study the microstructural changes of the multilayer, X-ray diffraction measurements of the samples with different d -spacings and thickness ratios were performed. An increased crystallization along with phase changes were observed in the samples prepared with thicker W layers, which can increase the compressive stress of the multilayer.

Keywords: X-ray, W/Si Multilayer, Interface Width, Stress, Crystallization.

1. INTRODUCTION

The X-ray Timing and Polarimetry (XTP) mission has been proposed in China for the study of astronomy physics under extreme gravity, density, and magnetism.¹ For this mission, X-ray focusing telescopes working in the energy region of 1–30 keV are designed and manufactured based on the Wolter-I type optical configuration.^{2,3} Each telescope consists of thousands of thin mirrors coated with depth-graded multilayers to improve the reflectivity in the high energy region. W/Si multilayers can provide high reflectivity with good stability in the high energy region, and these multilayers have been used in other previous missions.⁴ In the XTP mission, W/Si multilayers are used as reflective coatings for the telescopes. The stress of multilayers is a critical issue for the development of focusing telescopes, because it can distort the ultrathin mirrors and deteriorate the angular resolution.⁵ Thus, W/Si multilayers with high reflectivity, small interface widths (including

interface roughness and diffusion), and low stress need to be developed. The multilayer structure and the stress are affected by different structural parameters, including the thickness ratio, d -spacing, bilayer number, and so on. For instance, the stress of Mo/Si multilayers depended on the thickness ratio, and the stress value approached zero when γ_{Mo} (ratio of the thickness of Mo to the multilayer period) = 0.6.⁶ In the case of Mo/B₄C multilayers, the interface roughness increased with an increase in the number of the bilayer period.⁷ Similar studies have also been carried out using W/Si multilayers. The interface widths of the W/Si multilayer is affected by the microstructural changes associated with increase in the d -spacing. Transmission electron microscopy studies have indicated that the interlayer widths increased from 0.83/0.58 nm at $d = 3.43$ nm to 1.08/0.63 nm at $d = 6.86$ nm, respectively for the interfaces of W-on-Si and Si-on-W.⁸ Dhawan et al. found that the transition of tungsten layers from amorphous to crystalline state can cause an increase in the roughness of the tungsten layer.⁹ Windt studied the

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variation of W/Si multilayer stress as a function of the thicknesses of the individual layers of the multilayer¹⁰ and reported that the crystalline state of tungsten is an important factor that affects the multilayer stress. To understand the stress properties and the microstructure of the W/Si multilayers more comprehensively, and to provide guidance for the development of depth-graded multilayer mirrors for the XTP mission, we recently performed a series of experiments and characterizations based on the W/Si multilayer system. These include study of effects of different deposition conditions and post-annealing on the microstructure and stress of W/Si multilayers.^{11,12} In this paper, we have focused on the study of stress and microstructure of W/Si multilayers with respect to different structural parameters, including *d*-spacing, thickness ratio of tungsten (ratio of the thickness of W to the multilayer period), and bilayer number, which are the main optimizing parameters for the design of depth-graded multilayers. The effects of different parameters on the microstructure and the stress have been discussed based on the experimental results, and suitable multilayer structures with low stress and good interface have been proposed.

2. EXPERIMENTAL DETAILS

The W/Si multilayers were prepared by direct current (DC) magnetron sputtering using a dedicated deposition facility developed for cylindrical mirrors used in the telescope.¹³ The background pressure before deposition was 8×10^{-5} Pa. High purity argon (99.999%) was used as the working gas, and the working pressure was fixed at 0.4 Pa during the deposition. For this study, the multilayers were grown on flat substrates: 1 mm-thick and 30 mm-diameter polished quartz substrates (surface roughness of ~ 0.4 nm) for stress measurements, and 30 mm $\times$ 30 mm Si (100) wafers (surface roughness of ~ 0.2 nm) for structural characterizations. After the deposition, the samples were characterized by grazing incident X-ray reflection (GIXR) using an X-ray diffractometer with Cu K α radiation ($\lambda = 0.154$ nm) source. By fitting the GIXR curves using Bede Refs software (genetic algorithm), we can determine the individual W and Si layer thicknesses and the interface widths.¹⁴ X-ray diffraction (XRD) measurements were performed to analyze the crystalline states of the different samples.¹⁵ A laser interferometer was used to measure the radius of curvature of the quartz substrates before and after the coating, and the multilayer stress was calculated using the modified Stoney equation,

$$\sigma_f = (1/R_{\text{post}} - 1/R_{\text{pre}})(E_s/(1 - V_s))t_s^2/6t_f$$

where t_s is the substrate thickness, t_f is the film thickness, R_{pre} and R_{post} are the radius of curvature of the sample before and after deposition, respectively, and E_s and V_s are the Young's modulus and the Poisson's ratio of the substrate, respectively.¹⁶ For the stress measurement of the

Table 1. The layer structure parameters of all samples.

Group	1				2				3		
Number	1	2	3	4	5	2	6	2	7	8	
<i>D</i> -spacings (nm)	2.7	3.7	4.4	5.5	3.8	3.7	3.6	3.7	3.6	3.7	
Thickness ratio of W	0.45	0.46	0.47	0.47	0.30	0.46	0.67	0.46	0.46	0.45	
Bilayer number	80	80	80	80	80	80	80	80	120	160	

multilayer structure, three samples of each multilayer were prepared, and the average stress was calculated to represent the stress state. The error bar was calculated according to the stress values of the three samples. An atomic force microscope (AFM) was used to characterize the surface morphology of the multilayers.

Three groups of samples with different *d*-spacings, thickness ratios, and bilayer numbers were prepared. The first group consisted of four samples with the same thickness ratio of W layer, $\gamma_W = \sim 0.46$ and different *d*-spacings, $d = 2.7, 3.7, 4.4$, and 5.5 nm. These values were selected for the *d*-spacing because the thicknesses of depth-graded multilayers designed for the XTP mission are mainly in the range of 2.5 to 5.5 nm. The total number of bilayers, *N*, was 80. The total thickness of the multilayer is equivalent to the product of the *d*-spacing and the bilayer number. The second group of samples was prepared with the same *d*-spacing of ~ 3.7 nm and different thickness ratios of W layer, $\gamma_W = 0.3, 0.46$, and 0.67 . The total number of bilayers was 80. The third group of samples was prepared with different bilayer numbers, $N = 80, 120$, and 160 , with the same *d*-spacing of 3.7 nm and the same tungsten thickness ratio of 0.46 . The parameters of the layer structures of all the samples are listed in Table I.

3. RESULTS AND DISCUSSION

3.1. W/Si Multilayers with Different *d*-Spacings

The GIXR measurements and the fitted results of the 1st group samples are shown in Figure 1. For all the *d*-spacings, the layer densities of both W and Si were

Figure 1. GIXR curves of the W/Si multilayer samples with different *d*-spacing.

above 90% of the bulk value (bulk densities of W and Si are 19.30 and 2.33 g/cm³, respectively), while the average interface widths of W-on-Si and Si-on-W changed slightly, from 0.31 to 0.36 nm, as the d -spacing was increased from 2.7 to 5.5 nm. Owing to the uncertainty of the fitted results of the GIXR measurements, it is hard to differentiate the small difference in the interface widths of these multilayers at different d -spacings. Nevertheless, the interface widths of the multilayers prepared with different d -spacings are similar to the values reported by Christensen et al. for the Nustar mission (0.4 nm).⁴ Based on the above findings, the average interface widths of the depth-graded multilayer structure (with $\gamma_W = 0.46$) are expected to be approximately 0.31–0.36 nm.

The measured stresses of the different multilayers are shown in Figure 2. The compressive stress of the multilayer with a d -spacing of 2.7 nm is −73.3 MPa, and the stress increases monotonically to −465.5 MPa when the d -spacing is increased to 5.5 nm. The stress of the multilayer with $d = 5.5$ nm is similar to the value reported by Windt.¹⁰ The lower stress of the multilayer at smaller d -spacings is advantageous for the development of the depth-graded multilayer, since most of the layers in the multilayer stack have small thicknesses as designed by the Power-Law method.¹⁹ This structure is suitable for the preparation of a depth-graded multilayer with a relatively low stress.

To study the microstructural changes and the possible mechanisms for the stress evolution, the samples were characterized using XRD, and the results are shown in Figure 3. The multilayer with a d -spacing of 2.7 nm is in an amorphous state, since there is no observable diffraction peak. As the d -spacing is increased to 3.7 nm, the tungsten layer begins to crystallize. However, the diffraction peak is very broad and weak, and therefore, most of the layers are nearly amorphous. When the bilayer thickness is increased to 4.4 and 5.5 nm, the crystallization is enhanced, and the center position of the peak shifts from 39.1 to 38.6°. To further analyze the change in the crystallization, the XRD

Figure 2. The stress changed with the increasing d -spacing.

Figure 3. The XRD measurements of the different d -spacing samples.

profiles were fitted appropriately. Three different cubic crystalline structures of tungsten have been reported in the literature for thin films. Among these, the most stable structure is α -W (space group Im-3m), and the other two structures, β -W (space group Pm-3n) and γ -W (space group Fm-3m), are metastable.^{17,18} The α and the β phases are well discussed in the literature, while the γ phase is not widely reported. As discussed in Ref. [18], the γ -W phase is observed only in thin films grown under particular conditions. For example, it has been reported to grow on silicon oxide, which is similar to the present case. The XRD profiles of all the samples were fitted using the three different phases of W, namely α -W(110) ($2\theta = 40.26^\circ$), γ -W(111) ($2\theta = 38.37^\circ$), and β -W(200) ($2\theta = 35.52^\circ$). The crystalline phases of α -W(110) and β -W(200) were also observed in the W/Si multilayers reported by Windt et al.¹⁰ The possible phases of various W_xSi_y compounds at the interfaces were not taken into account for the fitting, since the interface width of the multilayer is very small and the contribution is negligible. The fitted results are shown in Figure 4. At a d -spacing of 3.7 nm, the multilayer consists of predominantly the α -W(110) phase, along with small amounts of the γ -W(111) phase. The diffraction peak of W(200) is almost unobservable. As the d -spacing is increased to 4.4 nm, the intensities of all the three phases increase; however, the α -W(110) peak still exhibits the highest intensity. The α -phase of W usually forms at low pressure deposition conditions and exhibits a high compressive stress,^{10,23,24} which is consistent with the results of the present study. As the d -spacing was increased to 5.5 nm, the intensities of all the three phases further increase, but the γ -W(111) and the α -W(110) phases are dominant. This is also indicated by the shift in the positions of the measured peaks to smaller angles. Therefore, it can be considered that the preferential crystallization of tungsten changed at a larger layer thickness. The enhanced polycrystallization at larger d -spacings did not affect the interface widths of the multilayers significantly, as indicated by the GIXR results. The evolution of the layer structure can lead to stress changes in the multilayer. For the

Figure 4. The multi-peak fitting of the XRD measurement. (a) $d = 3.7$ nm, (b) $d = 4.4$ nm, (c) $d = 5.5$ nm.

amorphous multilayer with a small d -spacing of 2.7 nm, the interfacial stress is considered to be predominant, especially since the growths of W layer on Si and Si layer on W in the multilayer can result in inverse stress states.²¹ This resulted in a small stress in the sample prepared with a d -spacing of 2.7 nm. At a larger d -spacing, the crystallization of the W layers increased, and the strain from the α -W and probably the γ -W grains increased. This can result in an increase in the compressive stress of the multilayers prepared with d -spacings of 4.4 and 5.5 nm. Another possible reason for the increase in the stress with increase in the d -spacing is the growth of thicker layers in each period. At a larger d -spacing, the additional Si and W layers grown after the initial growth stage at the

interface can induce compressive stresses because of the atomic peening effect, which is due to the bombardment of the deposited film by particles with a relatively high kinetic energy and densification of the film.^{25,26}

3.2. W/Si Multilayers with Different Thickness Ratios

The second group of samples were prepared with different thickness ratios of tungsten, $\gamma_W = 0.3, 0.46$, and 0.67 . In this range, the thickness ratio can not only affect the multilayer stress, but also change the reflectivity of the multilayer. Theoretically, a smaller thickness ratio of W can reduce the absorption of X-rays and result in a higher average reflectance of the depth-graded multilayer. Thus, both the stress and the theoretical reflectivity have to be considered for the optimization. The GIXR measurements and the fitted results are shown in Figure 5. The shifts in the peak positions of the three samples are caused by the small difference in the multilayer d -spacing. According to the fitted results, there is only a slight change in the average interface width, from 0.33 to 0.36 nm, with increase in the thickness ratio of tungsten from 0.30 to 0.67, and the layer densities of both W and Si are greater than 90% of the bulk values.

The results of stress measurements of the samples are shown in Figure 6. The multilayer with $T_W = 0.46$ exhibited a compressive stress of approximately -265 MPa. As the thickness ratio was decreased or increased to 0.30 and 0.67, respectively, the stresses increased to approximately -300 MPa in both the cases. Among the three samples repeated for each thickness ratio, the largest value of stress in the samples with $T_W = 0.46$ is still smaller than the smallest value of the stress in the samples with $T_W = 0.30$ and 0.67. This implies that the multilayer with $T_W = 0.46$ seems to be the smallest stress state within the experimental error. Considering the difference in both the stress and the average reflectivity of the depth-graded multilayer, the optimal thickness ratio of W is considered to be $T_W = 0.4-0.5$.

Figure 5. The GIXR curves of the W/Si multilayer samples with different thickness ratio of W.

Figure 6. The stress changed with the increasing thickness ratio of W.

The XRD patterns of the samples are shown in Figure 7. For the multilayer with $\Gamma_W = 0.30$ ($d_W \approx 1.1$ nm), no diffraction peaks were detected, and therefore, the layers are amorphous. As the thickness ratio is increased from 0.30 to 0.67, the intensities of the diffraction peaks gradually increase, especially from $\Gamma_W = 0.46$ to $\Gamma_W = 0.67$. The center position of the peak shifts from 40.17° to 39.11° . The XRD curves were also fitted assuming the three phases of elemental tungsten, namely α -W(110), γ -W(111), and β -W(200). As the multilayer with $\Gamma_W = 0.46$ is the same sample as that in the 1st group with $d = 3.7$ nm, the fitted results of only the multilayer with $\Gamma_W = 0.67$ are shown in Figure 8. It can be seen that the multilayer mainly consists of α -W(110) and γ -W(111), while the crystallization of W(200) is relatively weak. This is similar to the result obtained for the sample with $d = 4.4$ nm in the 1st group, which has a similar W layer thickness. The stress changes with different thickness ratios can be explained using the similar interpretation provided for the samples in the 1st group. For the amorphous multilayer with $\gamma_W = 0.30$, the interface stress can be dominant, while the thicker Si layer grown on W seems to contribute to the larger compressive stress as compared to the sample with

Figure 7. The XRD measurements of the different thickness ratio samples.

Figure 8. The multi-peak fitting of the XRD measurement of $\gamma_W = 0.67$.

$\gamma_W = 0.46$. This compressive stress state of Si was also observed in WSi_2/Si ²² and Mo/Si multilayer systems.⁶ At a large thickness ratio of $\gamma_W = 0.67$, the obvious polycrystallization of the W layers in the α - and the γ -phases may increase the compressive stress, which gradually exceeds the interface effects, resulting in a relatively large stress state.

3.3. W/Si Multilayers with Different Bilayer Numbers

The GIXR measurements and the fitted results of the multilayers prepared with different numbers of bilayers are shown in Figure 9. When the bilayer number is increased from 80 to 160, the average interface width is almost unchanged at ~ 0.35 nm. The surface morphology of the multilayers with 80 and 160 bilayers were studied by AFM, and the results are shown in Figure 11. Both the samples exhibited almost the same surface roughness with root-mean-square roughness values of 0.20–0.18 nm, and peak-to-valley heights of 1.9–1.5 nm. These values are consistent with the interface widths fitted from the GIXR results. The surface morphology of the two samples were also similar, which indicates that the layer growth process

Figure 9. The GIXR curves of the W/Si multilayer samples with different bilayer number.

Figure 10. The stress is unchanged with the increasing bilayer number.

remains the same from 80 to 160 bilayers. No accumulation of roughness was found with increase in the bilayer number. This is important for the depth-graded multilayers, since a large number of bilayers, typically above 100, is usually needed to obtain a broadband response with a high integral flux and a relatively flat reflectivity profile.

The results for stress measurements are shown in Figure 10. The multilayer stress is unchanged with increase in the number of bilayers, with a value of approximately -265 MPa. As the stress calculated using the Stoney equation is normalized by the total thickness of the multilayer, the constant value of stress at the different bilayer numbers implies that the microstructures of the

multilayers are unchanged when N is increased from 80 to 160. The force in the film per unit width, F/w , can be calculated based on the stress given by the Stoney equation as $F/w = \sigma_f \cdot t_f$, as indicated in Ref. [27]. It was found that the force generated in the multilayer increased from -78.4 to -156.8 N/m as the bilayer number was increased from 80 to 160. This means that the total amount of distortion caused by the multilayer stress increases linearly with increase in the number of the bilayers.

4. CONCLUSION

The stress and the layer structure of W/Si periodic multilayers with different d -spacings, thickness ratios, and bilayer numbers were studied to develop X-ray mirrors for the proposed XTP mission. It was found that the average interface width changed insignificantly, from 0.31 to 0.36 nm, with increase in the d -spacing from 2.7 to 5.5 nm, and the stress increased from -73.3 to -465.5 MPa. For the multilayers prepared with different thickness ratios and the same d -spacing of ~ 3.7 nm, the lowest stress of -265 MPa was observed at $T_w = 0.46$. Both the average stress and the interface widths were unchanged with increase in the number of bilayers from 80 to 160. The changes in the stress are believed to be due to a combination of several factors, such as the interfacial stress, crystallization of the W layers, and atomic peening effect. These results are expected to provide important guidance for the optimization of structural parameters during the design and fabrication of W/Si multilayer mirrors with a low stress and a relatively high reflectivity.

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References and Notes

1. Y. Dong, *Proc. SPIE* 9144, 91443O-1 (2014).
2. S. L. O'Dell, R. J. Brissenden, W. N. Davis, R. F. Elsner, M. S. Elvis, M. D. Freeman, T. Gaetz, P. Gorenstein, M. V. Gubarev, D. Jerius, M. Juda, J. J. Kolodziejczak, S. S. Murray, R. Petre, W. Podgorski, B. D. Ramsey, P. B. Reid, T. Saha, D. A. Schwartz, S. Trolier-McKinstry, M. C. Weisskopf, R. H. T. Wilke, S. Wolk, and W. W. Zhang, *Phys.* 7803, 373 (2010).
3. W. W. Zhang, *Proc. SPIE* 8147, 309 (2011).
4. F. E. Christensen, A. C. Jakobsen, N. F. Brejnholt, K. K. Madsen, A. Hornstrup, N. J. Westergaard, J. Momberg, J. Koglin, A. M. Fabricant, W. W. Craig, M. J. Pivovarov, and D. Windt, *Proc. SPIE* 8147, 309 (2011).
5. K. W. Chan, M. Sharpe, W. Zhang, L. Kolos, M. Hong, R. McClelland, B. Hohl, T. Saha, and J. Mazzarella, *Proc. SPIE* 8861(s 1–3), 564 (2013).
6. S. S. Andreev, N. N. Salashchenko, L. A. Suslov, A. N. Yablonsky, and S. Y. Zuev, *Nucl. Instrum. Methods Phys. Res., Sect. A* 470, 162 (2001).

Figure 11. (a) The AFM measurement of $N = 80$. (b) The AFM measurement of $N = 160$.

7. H. Wang, D. Xu, J. Zhu, Z. Zhang, R. Alnaimi, B. Mu, Z. Wang, and H. Chen, *OPTIK* 125, 3415 (2014).
8. D. L. Windt, F. E. Christensen, W. W. Craig, C. Hailey, F. A. Harrison, M. Jimenez-Garate, R. Kalyanaraman, and P. H. Mao, *J. Appl. Phys.* 88, 460 (2000).
9. R. Dhawan and S. Rai, *AIP Conf. Proc.* (2016), Vol. 1731, pp. 8151–1025.
10. D. L. Windt, *J. Vac. Sci. Technol. A* 18, 980 (2000).
11. R. Qi, Q. Huang, Y. Yang, Z. Zhang, and Z. Wang, *Opt. Eng.* 56, 035103 (2017).
12. Q. Huang, J. Zhang, R. Qi, Y. Yang, F. Wang, J. Zhu, Z. Zhang, and Z. Wang, *Opt. Express* 24, 15620 (2016).
13. C. P. Jensen, K. K. Madsen, H. C. M. Chen, and E. Ziegler, *Proc. SPIE* 4851, 723 (2003).
14. M. Wormington and D. K. Bowen, *Philos. T. Roy. Soc. A* 357, 2827 (1999).
15. A. L. Patterson, *Phys. Rev.* 56, 978 (1939).
16. G. G. Stoney, *P. Roy. Soc. Lond.* 82, 40 (1909).
17. K. Murugan, S. B. Chandrasekhar, and J. Joardar, *Int. J. Refract. Met. H* 29, 128 (2011).
18. J. I. Watjen, T. J. Bright, Z. M. Zhang, C. Muratore, and A. A. Voevodin, *Int. J. Heat Mass Tran.* 61, 106 (2013).
19. K. D. Joensen, P. Voutov, A. Szentgyorgyi, J. Roll, P. Gorenstein, P. Høghøj, and F. E. Christensen, *Appl. Optics* 34, 7935 (1995).
20. M. Wormington, C. Panaccione, K. M. Matney, and D. K. Bowen, *Phil. Trans. R. Soc. Lond. A* 357, 2827 (1999).
21. C. Liu, R. Conley, and A. T. Macrander, *Proc. SPIE* 6317, 63170J-1 (2006).
22. M. Kimberly, S. Bing, C. Ray, and T. M. Albert, *Appl. Phys. Lett.* 99, 7013 (2011).
23. B. Girault, D. Eyidi, P. Goudeau, T. Sauvage, P. Guerin, E. Le Bourhis, and P.-O. Renault, *J. Appl. Phys.* 113, 174310 (2013).
24. I. Djerdj, A. M. Tonejc, A. Tonejc, and N. Radić, *Vacuum* 80, 151 (2005).
25. D. M. Mattox, *Handbook of Physical Vapor Deposition (PVD) Processing*, Second Edition, Noyes Publications, Westwood (1998).
26. L. B. Freund and S. Suresh, *Thin Film Materials*, Cambridge University Press, Cambridge (2003).
27. J. M. Freitag and B. M. Clemens, *Appl. Phys. Lett.* 73, 43 (1998).
28. D. L. Windt, W. L. Brown, C. A. Volkert, and W. K. Waskiewicz, *J. Appl. Phys.* 78, 2423 (1995).
29. H. W. Schnopper, P. Gorenstein, and J. L. Wood, *Proc. SPIE* 1736, 239 (1993).
30. J. H. Underwood and T. W. Barbee, *Nature* 294, 429 (1981).
31. E. Majkova, S. Luby, M. Jergel, Y. Chushkin, E. D'Anna, A. Luches, M. Martino, P. Mengucci, G. Majni, Y. Kuwasawa, and S. Okayasu, *Appl. Surf. Sci.* 208, 394 (2003).
32. T. Salditt, D. Lott, T. H. Metzger, J. Peisl, G. Vignaud, P. Høghøj, O. Schärpf, P. Hinze, and R. Lauer, *Phys. Rev. B* 54, 5860 (1996).
33. C. Montcalm, *Opt. Eng.* 40, 469 (2001).
34. V. I. Pinegyn, E. N. Zubarev, V. V. Kondratenko, V. A. Severyukova, S. A. Yulin, T. Feigl, and N. Kaiser, *Thin Solid Films* 516, 2973 (2008).
35. K. K. Madsen, C. P. Jensen, E. Ziegler, K. S. Gunderson, and J. E. Koglin, *Proc. SPIE* 5168, 41 (2004).
36. C. P. Jensen, H. C. M. Chen, and E. Ziegler, *Proc. SPIE* 4496, 104 (2002).

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