Determination of the deposition rate of DC magnetron sputtering in fabrication of X-ray supermirrors

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X-ray supermirror is a non-periodic multilayer structure, whose optical performance is greatly affected by the stability and accuracy of the deposition rate in the fabrication using the direct current (DC) magnetron sputtering. By considering the location-setting time of the substrate positioning above the sputtering target, the deposition rate can be accurately determined. Experimental results show that the optical performance of the supermirror is in agreement with the design aim, which indicates that the layer thickness is well controlled and coincides with the desired ones.

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In the hard X-ray region, the optical elements based on the traditional periodic multilayers cannot meet with the requirement of the astronomical telescope, for their narrow response bandwidth in reflectance. One solution of widening the bandwidth of reflectivity may be found in a standard neutron optics device: the supermirror. Joensen *et al.* proposed the concept of X-ray supermirrors from the depth-graded multilayer based on neutron supermirrors[1]. Theoretically, X-ray supermirror is a non-periodic multilayer with the flat and high response reflectivity in wide energy or angle range. That is to say, each layer thickness is different in the multilayer stack[2,3]. Compared with multilayers produced for visible light, X-ray supermirrors have thickness of about a factor of 100 smaller and about 10 times more layers. Its layer thickness is usually on the nanometer or sub-nanometer scale. Therefore, it is most important to deposit and measure ultra-thin multilayer accurately. The magnetron sputtering, for its flexibility, stability and control of process conditions, has been used to fabricate supermirrors. The sputtering rate is affected by many factors such as power of the sputtering cathode, working gas flow, working pressure, vertical distance between substrate and sputtering target, and so on. Once the optimal experimental condition is confirmed, almost all the parameters are confirmed. The error introduced by the little change of the parameters is too weak to be considered. Some work has been reported on predicting deposition rate in the view point of microcosmic movement of sputtering particles[4−10], which is difficult to carry out directly in the fabrication experiments. A modified experiment method was described in the paper to calculate the deposition rate accurately by considering the movement of the substrate positioning above the sputtering source.

Figure 1 shows a schematic of a multi-target magnetron sputtering system (JGP560C6, China), which will be used to fabricate the tungsten/carbon (W/C) multilayer supermirrors. This sputtering system has four conventional circular sputtering cathodes, placed around the cylindrical chamber bottom and spaced 90° apart (just two sputtering cathodes are shown in Fig. 1). Each sputtering source can be connected with direct current (DC) or radio frequency (RF) source. All of the sputtering sources are of 100 mm in diameter. The general metal, non-metal materials (Mo, W, Si, C, B_4C, and so on), and the ferromagnetic materials (Fe, Co and Ni) can be deposited. The sputtering sources are separated by baffle plates in order to avoid cross-contamination during deposition. A water cooling system is used to avoid cracking of the targets due to temperature differences, and the chamber was pumped down to a base pressure of 1×10^{-4} Pa prior to the deposition. High purity Ar (99.999%) is used as working gas. On the upward side of the chamber, four substrate holders are mounted on a grounded table. The substrates are fixed upside-down on the rotating holders above the sputtering sources. In this paper, one substrate, labeled A, and two sputtering sources, labeled W (represented tungsten) and C (represented carbon) in Fig. 1, were used to deposit W/C supermirrors. During the magnetron sputtering process, a plasma discharge is maintained between the grounded rotation table (anode) and the sputtering targets (connected with sputtering sources, called sputtering cathodes). The vertical distance between substrate and sputtering targets can continuously be adjusted in the distance range from

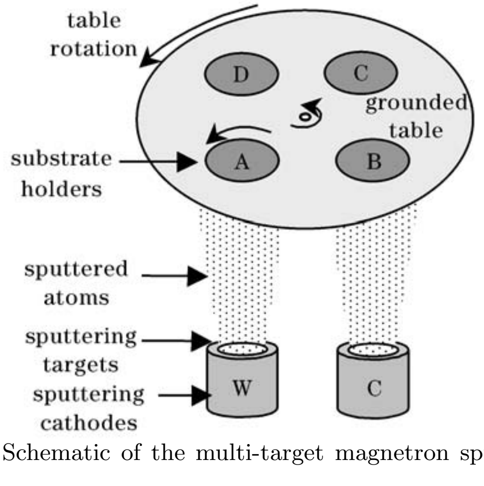

Fig. 1. Schematic of the multi-target magnetron sputtering system.

30 to 200 mm. The rotation of the grounded table ensures that one bilayer is deposited per revolution. When the substrate moves above the desired sputtering source, the location of the grounded table is fixed and waits for depositing. At the same time, the substrate holder also rotates around its axis to improve the uniformity of the multilayer. The individual layer thickness is determined by the stay time of the grounded table positioning above the sputtering source. Then, the substrate moves to next sputtering source with the revolution of the grounded table. In our experiments, the experimental parameters, such as the stay time of substrate above the desired sputtering source and the rotating velocity of grounded table, were controlled by a computer program. The W/C multilayers were fabricated onto silicon substrate (20 × 30 (mm)). The distance between each sputtering source and the substrate was 100 mm. The sputtering powers were 20 and 120 W for the targets of W and C, respectively. The revolution velocity of grounded table was 2 circles per minute, and the substrate rotated around its center at the speed of 10 circles per minute.

After fabrication, an X-ray diffractometer (XRD) (D1 system, Bede Inc., UK, working at 8.0 keV, the K_α line of Cu) provides the characterization of the period layer thickness and the optical performances of the multilayer supermirrors.

Figure 2 shows the designed and measured reflectivities of the W/C supermirrors. Curve (a) in Fig. 2 shows a designed non-periodic W/C supermirror working at the energy of 8.0 keV with a broad angular range. The calculated reflectivity is 0.245 ± 0.006 in the grazing incident angle range of $1.09° - 1.39°$. The layer thickness distribution of the designed W/C supermirror is shown in Fig. 3. It can be seen that the layer thicknesses are different for each W and C layers, which oscillate between of 1.8—3.1 nm for C layer and 0.8—1.8 nm for W layer, respectively. After design, this W/C supermirror will be fabricated using the DC magnetron sputtering system described above. In order to control each layer thickness using the depositing time, the deposition rate should be determined firstly.

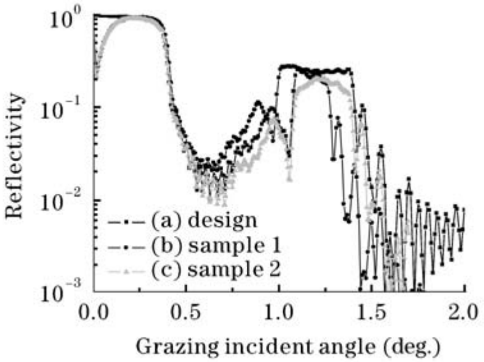

Fig. 2. Designed and measured reflectivities of the W/C supermirrors. Curve (a) is the designed result in the grazing angle range of $1.09° - 1.39°$ with the flat reflectivity of 0.245 ± 0.006. Curves (b) and (c) are the measured results of samples 1 and 2, respectively. Curve (b) shows that measured reflectivity of the sample 1 working in the grazing angle range of $0.98° - 1.28°$ with the mean reflectivity of 0.222 ± 0.055. Curve (c) shows the measured reflectivity of the sample 2 working in the grazing angle range of $1.09° - 1.39°$ with the mean reflectivity of 0.173 ± 0.020.

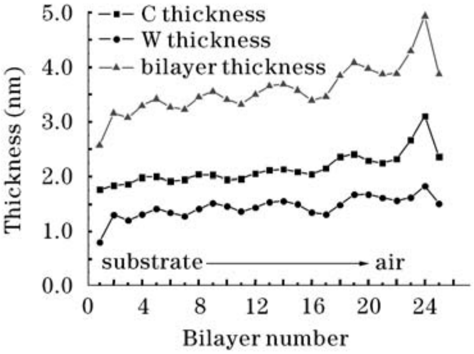

Fig. 3. Layer thickness distribution of the W/C supermirror designed for the grazing incident angle range of $1.09° - 1.39°$.

A simple model[11] has been developed to predict the deposition rate by calculating the combination of two periodic multilayers using the following equations:

$$\left\{ \begin{array}{l} v_{\rm W} t_{\rm W1} + v_{\rm C} t_{\rm C1} = d_1 \\ v_{\rm W} t_{\rm W2} + v_{\rm C} t_{\rm C2} = d_2 \end{array} \right. , \qquad (1)$$

where $v_{\rm W}$ and $v_{\rm C}$ are the deposition rates for the sputtering targets of W and C, respectively; $t_{\rm W1}$ ($t_{\rm W2}$) and $t_{\rm C1}$ ($t_{\rm C2}$) are the stay time of substrate over the sputtering sources of W and C for each revolution of grounded table. Given the different stay time $(t_{\rm W1}, t_{\rm C1})$ and $(t_{\rm W2}, t_{\rm C2})$, two types of W/C periodic multilayers can be fabricated with two period thicknesses of d_1 and d_2, which will be measured by XRD[12−15]. It should be noted that the matrices of $(t_{\rm W1}, t_{\rm C1})$ and $(t_{\rm W2}, t_{\rm C2})$ must be linearly independent. So, the deposition rates of the sputtering targets of W and C, $v_{\rm W}$ and $v_{\rm C}$, can be determined by calculating Eq. (1). In the experiment, the calculated results are $v_{\rm W} = 0.044$ nm/s and $v_{\rm C} = 0.038$ nm/s, respectively. Then, the designed W/C supermirror was fabricated (named sample 1) using these deposition rates and the layer thickness distribution presented in Fig. 3.

After fabrication, the reflectivity of the W/C supermirror was characterized by XRD. The measured reflectivity, as shown in curve (b) of Fig. 2, is 0.222 ± 0.055 in the angle range of $0.98° - 1.28°$, which deviates from the designed angle range of $1.09° - 1.39°$ by 0.11°. It indicates that the fabricated multilayer structure is not in agreement with the designed one. The possible reason is the deviations of the layer thicknesses. When the substrate moves in or out of the plasma rings above the sputtering sources in each revolution of the grounded table, this deposition process is completely neglected in the calculation described above, in fact, which is not ignored for fabrication of ultra-thin multilayer. So, the calculation precision of the deposition rate can be improved by considering the location-setting process of the grounded table positioning above the sputtering sources. Here, the location-setting time, Δt, includes processes of the substrate moving in and out of the plasma rings. In order to simplify the calculation, the deposition rates are regarded as constant, and the time of moving in and out of the plasma rings is equal for both sputtering sources. Equation (1) can be rewritten as

$$\begin{cases} v_{\mathrm{W}}(t_{\mathrm{W}1}+\Delta t)+v_{\mathrm{C}}(t_{\mathrm{C}1}+\Delta t)=d_1 \\ v_{\mathrm{W}}(t_{\mathrm{W}2}+\Delta t)+v_{\mathrm{C}}(t_{\mathrm{C}2}+\Delta t)=d_2 \\ v_{\mathrm{W}}(t_{\mathrm{W}3}+\Delta t)+v_{\mathrm{C}}(t_{\mathrm{C}3}+\Delta t)=d_3 \end{cases} . \quad (2)$$

Similar to the method described above, given three linear independent matrices of $(t_{\mathrm{W}1}, t_{\mathrm{C}1})$, $(t_{\mathrm{W}2}, t_{\mathrm{C}2})$ and $(t_{\mathrm{W}3}, t_{\mathrm{C}3})$, three types of W/C periodic multilayers can be fabricated with three period thicknesses of d_1, d_2 and d_3 being measured by XRD, and the deposition rates of the sputtering targets of W and C, v_{W} and v_{C}, and the location-setting time Δt can be determined. They are $v_{\mathrm{W}} = 0.047$ nm/s, $v_{\mathrm{C}} = 0.040$ nm/s, and $\Delta t = 3.0$ s, respectively. Using these deposition rates, another W/C multilayer supermirror was fabricated (named sample 2). Its reflectivity was also characterized by XRD. The measured reflectivity, curve (c) shown in Fig. 2, is 0.173 ± 0.020 in the angle range of $1.09^\circ - 1.39^\circ$, which coincides with the designed one.

It can be found that the revolution of the grounded table affects the coating process and the determination of the deposition rate. In fact, the location-setting time is not equal for the sputtering targets of W and C, because the sizes of plasma rings vary with the sputtering materials and condition. Moreover, the deposition rate cannot be regarded as constant within the plasma rings, especially during the location-setting process. Further work is required to investigate the influence of the location-setting process.

In the fabrication of ultra-thin multilayer using magnetron sputtering, the location-setting process of the grounded table positioning above the desired sputtering source cannot be neglected, which was found to influence the layer thickness and the determination of the deposition rate. And the optical performance of the fabricated multilayer is greatly affected by the deviation of deposition rate. In order to fabricate the multilayer with the designed layer thickness, a modified method, which takes into consideration of location-setting time, is described to determinate the deposition rate more accurately. The methods' result coincides with the experiment very well. In addition, this method is also applicable to calculate the deposition rate for the traditional periodic multilayer and other sputtering material pairs.

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