Effects of film thickness and annealing temperature on the properties of molybdenum carbide films prepared using pulsed direct-current magnetron sputtering

Zhenqing Wu1,2, Jiaoling Zhao1,2,3, Meiping Zhu1,2,3, Sheng Guo1, Tianbao Liu1,2, Wenyun Du1,2, Jun Shi1,2, Tingting Zeng1,2 and Jianda Shao1,2,3

1 Laboratory of Thin Film Optics, Key Laboratory of Materials for High Power Laser, Shanghai Institute of Optics and Fine Mechanics, Shanghai 201800, People’s Republic of China
2 Hangzhou Institute for Advanced Study, University of Chinese Academy of Sciences, Hangzhou 310024, People’s Republic of China
3 Center of Materials Science and Optoelectronics Engineering, University of Chinese Academy of Sciences, Beijing 100049, People’s Republic of China
4 Tongji University, MOE Key Laboratory of Advanced Micro-Structured Materials, School of Physics Science and Engineering, Shanghai 201800, People’s Republic of China

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Abstract

Molybdenum carbide (Mo2C) films were prepared using pulsed direct-current (DC) magnetron sputtering. The effects of film thickness on the phase structure, surface morphology, and optical constants of the films were examined using X-ray diffraction (XRD), atomic force microscopy, and extreme ultraviolet reflectivity. XRD analysis showed that the as-sputtered films with thicknesses between 30 nm and 150 nm were almost amorphous. New phase \( \alpha \)-MoC\(_{1-x} \) with the \( \{111\}, \{200\}, \{220\}, \{311\}, \{222\} \) crystal planes appeared in films with thicknesses between 200 nm and 400 nm. The phase transition and chemical composition of the Mo2C films treated using vacuum heat treatment were analyzed in detail. For 150 nm-thick films, the as-deposited as well as the 600 °C-annealed films were amorphous. The phase structures of the 150 nm-thick film annealed at 700 °C, 750 °C, and 800 °C were orthogonal Mo2C(\( \alpha \)-Mo2C), multiphase structure (\( \beta \)-Mo2C, monoclinic MoO\(_2\), and cubic MoN), and monoclinic MoO\(_2\), respectively. X-ray photoelectron spectrometry revealed that the Mo-Mo bonds of the films transformed into Mo-C, Mo-O, and Mo-N bonds under 750 °C-annealing, further confirming the formation of a multiphase structure after annealing. Thus, film thickness and annealing temperature considerably influence the properties of Mo2C films.

1. Introduction

Molybdenum carbide (Mo2C) films have attracted extensive attention owing to its advantages of high melting point, high hardness, excellent thermal stability, and mechanical properties [1]. Mo2C films have been widely used in various fields, such as catalysis [2–4], superconductivity [5, 6], and Bragg mirrors [7–9]. The applications of Mo2C films are largely dependent on the phase transitions, especially for use as catalysts. Walden et al studied the synthesis of molybdenum oxide films for application as hydrogen dissociation catalysts through plasma-enhanced chemical vapor deposition. Molybdenum oxide was completely transformed into \( \beta \)-Mo2C when heated to 700 °C under a mixture of 20% CH\(_4\) in H\(_2\) [2]. Wan et al prepared multiple phases of molybdenum carbide (\( \alpha \)-MoC\(_{1-x} \), \( \beta \)-MoC\(_{1-x} \), \( \eta \)-MoC, and \( \gamma \)-MoC) as electrocatalysts for the hydrogen evolution reaction (HER); they found that \( \gamma \)-MoC has great potential as catalyst for HER [3]. Yin et al synthesized porous \( \alpha \)-MoC\(_{1-x} \) nanorods via controlled carburization of MoO\(_3\) nanorod precursors, which exhibited significantly enhanced HER performance under both acidic and alkaline conditions [4]. Liu et al induced the transition from \( \alpha \) to \( \beta \)
phase in 2D α-Mo2C crystals using electron beam irradiation, they found that α/β Mo2C with sharp interfaces could improve superconductivity [5]. Xu et al investigated the fabrication of large-area high-quality 2D ultrathin superconducting α-Mo2C crystals using chemical vapor deposition (CVD); they found that the superconductivity was strongly dependent on crystal thickness [6]. In the field of extreme ultraviolet (EUV) reflective applications, Mo–Si intermetallic compounds and diffused interfaces form in a high-temperature environment, which leads to a decrease in the reflectivity and lifetime of EUV multilayer mirrors [10, 11]. Feigl et al found that the reflectivity of Mo/Si multilayers was significantly reduced after annealing above 300 °C, while Mo2C/Si multilayers maintained a superior thermal stability up to 400 °C [7]. Yuan et al prepared a Co/Mo2C multilayer mirror through magnetron sputtering, which showed that the multilayer was able to work up to 600 °C [8].

Studies have shown that the multiple phases of Mo2C films have different catalytic and superconducting effects; however, most of these preparation methods are CVD [2, 3, 6]. There are few detailed studies on the properties of molybdenum carbide films prepared using physical vapor deposition (PVD). In addition, the Mo2C can improve the thermal stability of multilayer films, Mo2C/Si-based mirrors have great potential for EUV lithography applications. Therefore, it is necessary to explore the microstructure and optical properties of Mo2C films prepared using PVD.

In this study, Mo2C films were prepared using pulsed direct-current (DC) magnetron sputtering and then vacuum-annealed. The effects of film thickness and annealing temperature on the phase transition, surface morphology, chemical composition, and EUV optical constants of the Mo2C films were explored. The EUV optical properties were analyzed, providing insights for the further preparation of Mo2C/Si-based mirrors in EUV applications.

2. Material and methods

2.1. Sample fabrication
Molybdenum carbide films with different thicknesses (10, 30, 60, 100, 150, 200, 300, and 400 nm) were deposited on a Si (110) substrate using pulsed DC magnetron sputtering. The films were sputtered from a round Mo2C target (purity, 99.95%, diameter = 100.0 mm, thickness = 6.0 mm). The target plane faced upward, and the substrate to be deposited faced downward. Si substrates (dimensions, 100 mm × 20 mm × 0.5 mm and 20 mm × 20 mm × 0.5 mm) were used for EUV reflectivity measurements and other characterizations, such as X-ray diffraction (XRD), atomic force microscopy (AFM), and X-ray photoelectron spectrometry (XPS). Prior to deposition, the chamber was pumped to a base pressure < 9.0 × 10−4 Pa. The Mo2C cathode was operated at 150 W. The deposition was performed under an Ar atmosphere of 2.5 × 10−3 Pa, and the deposition rate was 0.25 nm/s.

Molybdenum carbide films (thickness = 150 nm) were vacuum-annealed in a tube furnace at 600 °C, 700 °C, 750 °C, and 800 °C. Before annealing, the annealing furnace cavity was pumped to a base pressure < 1.0 Pa. During vacuum annealing, the temperature in the furnace was increased from 25 °C to the set temperature at a rate of 10 °C/min, kept constant for 1 h, and then allowed to cool to 25 °C naturally.

2.2. Sample characterization
The film thickness was characterized using a step profiler (Bruker Nano, DektakXT) and X-ray reflectometry (XRR; PANalytical Empyrean), the phase structures of the films were characterized using XRD (PANalytical Empyrean), and the grain size was calculated according to the Scherrer formula [12]. XRD patterns were obtained using CuKα radiation (λ = 0.154 nm), carried out at 40 mA and 40 kV, with a scanning angle range of 2θ = 20°–90° and a scanning step size of 0.05°. Surface morphologies of the prepared samples were characterized using AFM (Veeco Dimension 3100), performed in the contact mode, and field emission scanning electron microscopy (FE-SEM; Zeiss Auriga S40). The scanning range was 2.0 μm × 2.0 μm and 5.0 μm × 5.0 μm. The former was primarily used for analyzing local high-resolution morphologies, while the latter focused on analyzing the surface roughnesses of the samples. The chemical composition of the molybdenum carbide film (thickness = 150 nm) was determined using XPS (Thermo Scientific K-Alpha) with a monochromatic Al Kα (1486.6 eV) X-ray source. Before XPS scanning, the film was etched for 160 s with 1 keV Ar+ ions to remove surface contaminants. All XPS profiles were calibrated based on the binding energy of the C 1s line (284.8 eV). The EUV reflectivity was measured using the beamline of the synchrotron source at the Hefei National Synchrotron Radiation Laboratory.
3. Results and discussion

3.1. Effects of film thickness on the properties of the films

3.1.1 XRD analysis of the phase structures

Table 1 lists the designed thickness, measured thickness, and root-mean-square (RMS) roughness of the molybdenum carbide films. The standard deviation between the measured thickness and the designed thickness was approximately 3%; therefore, the designed thickness can be approximated as the actual thickness of the films. Figure 1 shows the XRD patterns of the films with different thicknesses. Compared with the XRD spectrum of Si (110), films with thicknesses of 10–150 nm only exhibited a substrate diffraction peak, which illustrates that the films are amorphous. For a greater thickness of 200 nm, the XRD diffraction peak was weak and wide, which was difficult to identify; the Si (110) diffraction peak also existed, which illustrates that the crystallization quality was poor. Studies [3] indicate that the standard α-MoC1-x usually has five diffraction peaks at approximately 36.41°, 42.30°, 61.36°, 73.50°, and 77.35° (JCPDS 01–089–2868), corresponding to the (111), (200), (220), (311), and (222) crystal planes, respectively. By comparing the measured diffraction peak position with the powder diffraction file (PDF) card (JCPDS 01–089–2868), the phase structure of the samples (200 nm, 300 nm, and 400 nm) was α-MoC1-x and the space group was Fm̅3 m, which is a face-centered cubic structure. Film growth takes the preferred orientation (111) crystal plane, and the preferred orientation is enhanced as the film thickness increases. Notably, these diffraction peaks are offset to the right by a certain angle relative to the standard diffraction peak positions. This can be explained by the presence of internal stress in the films [13].

The average grain sizes are calculated using the Scherrer formula:

\[ D = \frac{k \lambda}{\beta \cos \theta} \]  

(1)

where D is the average grain size (nm), k is the Scherrer constant (0.89), \( \lambda \) is the X-ray wavelength (0.154 nm), \( \beta \) is the diffraction peak half-width (FWHM), and \( \theta \) is the reflection angle. The grain sizes of the 200 nm, 300 nm, and 400 nm molybdenum carbide films are 9.1 nm, 11.4 nm, and 14.9 nm, respectively. Bae et al [14] have reported the similar result for TiNi shape-memory-alloy thin films deposited by magnetron sputtering, the grain size increased as film thickness increased and corresponded to the crystallinity. Takagi et al [15] reported that high-energy Ar⁺ ions can produce a thermal effect by bombarding the substrate during the deposition process.
Because the film thickness was controlled by the deposition time, increasing the sputtering time could improve the substrate temperature. This increase in the substrate temperature contributes toward the grain nucleation and growth [16]. From the perspective of thermal dynamics, an increase in the substrate temperature can promote the diffusion of deposited atoms during the growth of the crystal nucleus, which is conducive for grain growth. Therefore, the average grain size of the films increased with increasing film thickness.

3.1.2 AFM analysis of the surface morphology

Figure 2 shows the three-dimensional AFM surface morphologies of molybdenum carbide films with different thicknesses. Using three-dimensional topography, changes in the surface morphology and roughness were analyzed stereoscopically. RMS values are used to characterize the overall roughness. Figure 3 presents the measurement data and a fitting curve of the relationship between the RMS and film thickness; the error bar data are obtained using the standard deviation of the RMS. The small error bars indicate that the RMS of different points on the same sample have a lower difference. The fitting result fits the exponential function formula:

\[ y = 2.37 - 2.07 \times 0.9968^x \]  

where \( x \) is the thickness (nm), and \( y \) is the RMS (nm). The exponential function can quantitatively describe the relationship between surface roughness and film thickness. Surface roughness was significantly affected by the substrate temperature and atomic surface diffusion of the films. Potlog et al [17] have reported the similar behavior for NiO thin films deposited by magnetron sputtering, the changes in roughness with increasing film
Figure 4. Two-dimensional AFM morphologies of samples with different film thicknesses: (a)–(f) 30 nm, 100 nm, 150 nm, 200 nm, 300 nm, and 400 nm, respectively.

Figure 5. Calculated and experimental angle-dependent reflectivity of the samples at $\lambda = 13.5$ nm with different film thicknesses: (a)–(f) 30 nm, 100 nm, 150 nm, 200 nm, 300 nm, and 400 nm, respectively.
thicknesses are due to the growth evolution of the phase structures. The substrate temperature increased with increasing film thickness, which improved the diffusion ability of the deposited atoms and promoted grain growth. Therefore, the RMS value increased as the film thickness increased.

Figure 4 shows the two-dimensional AFM surface morphologies of the films with different thicknesses, with a scanning range of 2.0 μm × 2.0 μm. Özgür et al. [18] reported the formation of continuous thin films and the growing process of combining and merging with each other. It is clearly consistent with reported results, the combining process as shown in the figures 4(a)–(c) and the merging process as shown in figures 4(d)–(e). It is showed the whole film growth process which divided into two basic steps such as nucleation and crystallization. For thicknesses of 30–150 nm, the film was in the nucleation stage, and the crystal nucleus gradually grew up (figures 4(a)–(c)). With increasing film thickness, grain size gradually increased, further improving crystal quality. For thicknesses of 200–400 nm, uniformly distributed spherical grains grew. As shown in figures 4(e),(f), the grain sizes plateaued, which is related to the change in crystallinity. Thus, increasing film thickness promotes crystal growth, which is consistent with the results of the XRD crystallinity analysis, as shown in figure 1.

3.1.3 Analysis of EUV optical constant of the films The effects of film thickness on the optical constants were analyzed. The optical properties of the films can be described by the complex index of refraction (N),

\[ N = n + ik \]

where n is the refractive index, and k is the extinction coefficient, which is known as the optical constant. The accurate calculation of optical constants under specific technological conditions is crucial for the design and preparation of high-performance reflective mirrors. Angle-dependent reflectivity curves were measured at a wavelength of 13.5 nm using the beamline of the synchrotron source at the Hefei National Synchrotron Radiation Laboratory, and the range of the scanning incidence angle was 2°–90°. The optical constants were obtained by fitting angle-dependent reflectivity curves. Figure 5 shows the calculated EUV reflectivities of the films. The calculated curves are in good agreement with the measured ones, which indicates that the fitted optical constants are relatively accurate.
3.2. Effect of annealing temperature on the properties of the films

Table 2 lists the refractive indexes and extinction coefficients of the films with thicknesses of 30–400 nm obtained by fitting the angle-dependent reflectivity curves. The refractive indexes and extinction coefficients of molybdenum carbide films are 0.92820435 and 0.006951402, respectively. As shown in table 2, the film thickness does not have a significant effect on the optical constants. The refractive index increases slightly as the film thickness increases from 30 nm to 150 nm. The refractive index is closer to the standard in the database, and the extinction coefficient is comparatively smaller when the film thickness is 150 nm.

### 3.2. Effect of annealing temperature on the properties of the films

The core factors that affect phase structures are film thickness [19] and temperature [20]. According to the previous study, phase structure evolved with increasing film thickness. The phase structure of the as-deposited films (200, 300, and 400 nm) is α–Mo2C, and the films (30, 100, and 150 nm) is amorphous. We deduce that 150 nm is the critical thickness of possible crystallization for the as-deposited films. Therefore, the 150 nm films were used to study the effect of annealing temperature on the properties of the films.

#### 3.2.1. XRD analysis of the phase structures

Fils with a thickness of 150 nm were annealed at different temperatures to investigate the effect of the annealing temperature on the microstructure. As shown in figure 1, the 150 nm-thick film was amorphous before annealing. Figure 6 shows the XRD results of the annealed samples, corresponding to the XRD patterns at 600 °C (figure 6(a)), 700 °C (figure 6(b)), 750 °C (figure 6(c)), and 800 °C (figure 6(d)). As shown in figure 6(a), after annealing at 600 °C, the XRD pattern only includes a Si (110) diffraction peak, indicating that the structure is still amorphous. The standard α–Mo2C usually has three characteristic diffraction peaks at approximately 34.47°, 38.07°, and 39.53° (PDF 72–1683), corresponding to the (021), (200), and (121) crystal planes, respectively. As shown in figure 6(b), compared with the PDF card, the corresponding phase structure of the film annealed at 700 °C is α–Mo2C, and the space group is Pbcn. As shown in figure 6(c), the β–Mo2C, MoO2, and Mo2N phases coexist after annealing at 750 °C, with β–Mo2C as the main phase. Compared with the standard β–Mo2C (PDF 35–0787), the diffraction peaks at 34.56°, 37.49°, and 39.60° of the 750 °C-annealed film correspond to the (100), (002), and (101) crystal planes of β–Mo2C, respectively, where the space group is P63/mmc, and belongs to the hexagonal close-packed group. However, as β–Mo2C has a strong diffraction peak at approximately 37°, the diffraction peak corresponding to the (111) crystal plane of monoclinic MoO2 cannot be easily identified. In addition, the (−111) and (−222) crystal planes of MoO2 at approximately 26.14° and 53.77° have relatively better crystal orientations. The weak diffraction peaks at 37.45° and 75.87° correspond to the (111) and (311) crystal planes of standard cubic Mo2N (PDF 25–1366). As shown in figure 6(d), the 800 °C-annealed film has a dominant monoclinic MoO2 phase (PDF 76–1807) and a weak α–Mo2C phase (PDF 72–1683). The effect of annealing temperature on the phase structures of the samples with thicknesses of 30, 100, 200, 300, and 400 nm is analysed in supplementary materials.

![Figure 7](image)

Figure 7. Surface SEM images of 150 nm films annealed at different temperatures: (a)–(d) as-deposited, 700 °C, 750 °C, and 800 °C, respectively.
3.2.2. SEM analysis of the surface morphology

Figure 7 shows the surface morphologies of the 150 nm films before and after annealing. The surfaces of the as-deposited films (figure 7(a)) were relatively smooth and uniform. After annealing at 700 °C, 750 °C, and 800 °C in vacuum, the surface morphologies, as shown in figures 7(b)–(d), respectively, changed significantly compared with the as-deposited film, which was uneven and irregular. In addition, the gaps between the crystallites of the annealed films were large, likely due to the difference in the growth direction of the various phase structures, which is related to the heat-induced phase transition [21].

3.2.3. XPS analysis of the chemical composition

As shown in figures 8(a) and (b), Mo, C, N, and O are clearly identified from the survey of 150 nm molybdenum carbide films, where five distinct peaks can be observed around 228.4 eV, 284.9 eV, 394.6 eV, 412.1 eV, and 530.6 eV, corresponding to Mo 3d, C 1s, N 1s, Mo 3p, and O 1s, respectively. To further analyze the effect of heat treatment on the molybdenum carbide films, Mo 3d high-resolution spectra of the as-deposited and 750 °C-annealed were analyzed using Gaussian fitting in figures 8(c) and (d). The as-deposited films have fewer Mo-O bonds, indicating that the films are slightly oxidized during the deposition. For the bonding state of Mo, the Mo-Mo bonds of the as-deposited transformed into the Mo-C, Mo-O and Mo-N bonds of the 750 °C-annealed films, further confirming that the multiphase structure was formed after vacuum annealing. As shown in figure 8(d), the binding energies at 228.4 eV (Mo 3d5/2) and 231.54 eV (Mo 3d3/2) correspond to the Mo-N bond in the Mo2N phase, those at 228.6 eV and 231.9 eV correspond to the Mo-C bond in the β-Mo2C phase, and those at 229.3 eV and 232.6 eV correspond to the Mo-O bond in the Mo2O phase. These peak positions are consistent with reported Mo 3d results [21, 22]. The sp² C = C bond comprises a σ bond and a π bond, attributed to the double bond orbital hybrid, which can reduce friction during the rubbing process. The sp³ C–C only has a σ bond, which increases the hardness of the films [22, 23]. As shown in figures 8(e) and (f), the ratio of sp³/sp² reduced considerably after annealing, which significantly affected the mechanical properties and friction performance of the films [22]. Figure 8(f) presents the XPS peak spectrum of C 1s, which shows five peaks. The peak at the lower bond energy of 283.7 eV corresponds to the carbon in β-Mo2C, and the peaks at 284.7 eV,
285.6 eV, 287.3 eV, and 288.9 eV correspond to the four chemical bonds of \( \text{sp}^2 \text{C} = \text{C}, \text{sp}^3 \text{C}-\text{C}, \text{C}-\text{N}, \) and \( \text{C}-\text{O}, \) respectively [22, 23].

4. Conclusion

In this study, molybdenum carbide films with different thicknesses were prepared using pulsed DC magnetron sputtering and then vacuum-annealed. The effects of film thickness and annealing temperature on the properties of the molybdenum carbide films were examined. The phase structure of the as-deposited films (200, 300, and 400 nm) is \( \alpha-MoC_{1-x} \), with film growth occurring along the preferred direction of the (111) crystal plane, and the preferred orientation is enhanced with increasing film thickness. In addition, the RMS increased exponentially with increasing film thickness, and the increasing trend gradually decreased. The EUV optical constants were obtained by fitting the angle-dependent reflectivity curves. It is suggested that film thickness does not significantly affect the optical constants, and the \( n \) and \( k \) values of the molybdenum carbide films were approximately 0.9280 and 0.006208148 at a wavelength of 13.5 nm, respectively. The as-deposited and 600 °C-annealed molybdenum carbide films were amorphous. Phase transition occurred at approximately 700 °C. In addition, the phase structures of the 150 nm films annealed at 700 °C, 750 °C, and 800 °C corresponded to \( \alpha-MoC, \) the multiphase structure (\( \beta-Mo_{2}C, \) monoclinic MoO$_2$, and cubic Mo$_2$N), and the multiphase structure (monoclinic MoO$_2$ and \( \alpha-Mo_2C \)), respectively. The XPS analysis shows that the Mo-Mo bonds of the films transformed into the Mo-C, Mo-O, and Mo-N bonds after 750 °C annealing.

Insights about the effects of film thickness and annealing temperature on the properties of molybdenum carbide films are essential for acquiring the microstructural and optical characteristics of Mo$_2$C films. This study thus provides insights for the preparation of Mo$_2$C/Si-based multilayer structures for EUV applications.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

Disclosures

The authors declare no conflicts of interest.

ORCID iDs

Jiaoling Zhao © https://orcid.org/0000-0001-6733-090X

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