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ARTICLE

Etching of Low Energy Argon Ion Beam on Beryllium

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Abstract: The etching of low energy argon (Ar) ion beam of 0–1000 eV on Be was investigated. Different surface polishing methods were compared. Results show that the high-quality Be surface can be obtained by Ar ion beam etching. With the etching proceeding, the Be surface quality is gradually improved, and the surface roughness becomes stable, reaching 0.63 μm after etching at 600 eV and 100 mA for 6 h. White light interferometer (WLI) and focused ion beam (FIB) measurement methods were compared. Results show that FIB measurement method is more suitable for measurement of Be etching thickness. The experiment results and theoretical calculations suggest that the Be sputtering process is similar to the ionization process of Be by Ar ion bombardment. The influence law of Ar ion energy on sputtering yield of Be can be obtained with the first ionization energy as the sputtering threshold, and the variation of Be etching rate with the product of Ar ion beam energy and sputtering yield is obtained, providing foundation for engineering application of Be etching.

Key words: argon ion beam; beryllium; etching

Beryllium (Be) is an indispensable and valuable material in atomic energy, national defense, electronic products, telecommunications, and metallurgical industries[1], and it is mainly used in high-precision parts. Fabrication of Be part involves the complex structure manufacture with high machining accuracy[2] and high-quality Be surface[3]. With the rapid development of civilian space programs, the requirements for precise Be parts are increasing. The traditional machining methods and precise pattern machining can hardly achieve smooth Be surface. Ion beam etching is an important micromachining technique and ultra-precision machining method to strip the material surface. Due to the low temperature during ion beam treatment, the workpiece will not be subjected to thermal deformation and the as-processed surface is clean and smooth[4]. Ion beam etching and polishing techniques have been widely used to obtain the high-quality surface and accurate graphic etching[5].

Si film was treated by Ar ion beam etching on the surface of Be bulk to manufacture space mirror[6]. Keim et al[7] found that the ionization yield of Ar on Be surface is increased with increasing the temperature. Dobes et al[8] studied the interaction between nitrogen ions (5 keV) and the Be surface and found that 0.4 Be atom can be sputtered by one nitrogen atom. Zhao et al[9] investigated the X-rays induced by interactions of highly charged Ar ions with Be surface and reported that the etching effect of Ar ion beam with low energy on Be surface is still obscure. The outer electron layer of inert gas is fully filled, leading to the efficient electron scattering effect. Thus, the inert gas has excellent sputtering yield, and Ar is the most commonly used inert gas in the ion sputtering processes due to its low cost[10–11].

The interaction between Ar ion beam with low energy and Be is crucial in the ion beam etching process. In this research, the Be surface was polished and etched by Ar ion beam, and the influence of sputtering yield and etching rate of Ar ion beam on Be surface was studied, providing a basic support for the accurate etching on Be surface of complex structure.

1 Experiment

The raw specimen was Be bulk produced through vacuum hot-pressing (Ningxia Orient Group Co., Ltd, China). The measured density of Be was 1.83 g/cm^3 with error of 0.1%, and the measured purity of Be was over 99% (ICP-AES, Plasma 2000, NCS Testing Technology Co., Ltd, China). As shown in Table 1, Be specimens of five states were used in this research.

As shown in Fig.1, the Ar ion beam etching was conducted by the ion beam etching system (LKJ-3D-150, Beijing

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Table 1 Be specimens of different states

State	Treatment process
1	Be material was processed into Be bulk with thickness of 1 mm by turning
2	Be bulk of 1 mm in thickness was ground by 800#, 1200#, and 2400# sandpaper and polished by polishing agents of 3.0 and 1.5 μm in size
3	Polished Be bulk was electropolished in 5vol% aqueous hydrofluoric acid solution at 2.5 V for 5 s
4	Electropolished Be bulk was etched by ion beam with different voltages and currents
5	Etched Be bulk was processed into Be specimens with thickness of 0.5 mm by wire cutting, then ground by 800#, 1200#, and 2400# sandpaper until surface roughness was about 50 μm, and finally ground by standard flat crystal with diamond size of 6, 3, 1, and 0.5 μm until the surface roughness was about 30 μm

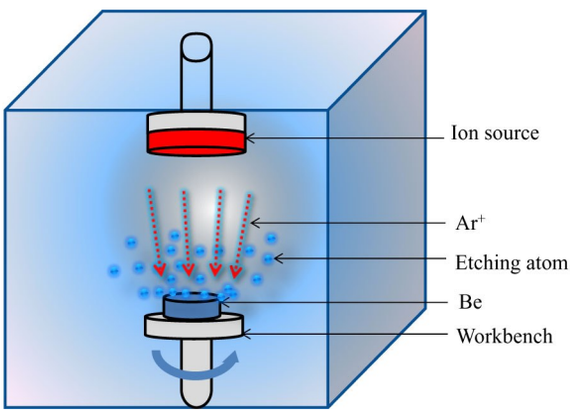

Fig.1 Schematic diagram of Ar ion beam etching on Be surface

Advanced MEMS Ion Beam Technology Research Institute Co., Ltd, China). The ion energy was 0–1000 eV. The peak beam density was larger than 1.0 mA/cm^2. The effective beam diameter was 100 mm. The tilting angle of the workbench was 0°–90°. The background pressure of the chamber was ⩽5×10^{-4} Pa. The stainless steel sheet with thickness of 0.015 mm was used as the mask plate.

Electron back-scattered diffraction (EBSD) was conducted by TEAM system of energy dispersive spectroscope (EDS, Oxford Ultim Extreme EDS, Britain), and the data processing was performed by TSL OIM Analysis 7. The morphologies before and after etching were observed by focused ion beam (FIB) system (FIB Helios Nanolab 600i, FEI Co., Ltd, America). The thickness and other dimension parameters were measured by FIB and white light interferometer (WLI, Talysurf CCI6000, Taylor Hobson Co., Ltd, Britain) methods. The surface roughness was measured by laser confocal microscope (LEXT OLS4100, Olympus Corporation, Japan). The specimen mass was measured by the precision balance (Cubis MSA6.6S-000-DM, Sartorius Company, Germany).

2 Results and Discussion

2.1 Effect of Ar ion beam etching on Be surface

Because Be surface after mechanical polishing, chemical polishing, electrolytic polishing, or other combined methods cannot satisfy the EBSD observation requirements, EBSD measurement cannot be well calibrated. As shown in Fig. 2a, the calibration rate of diffraction pattern is inferior after standard mechanical polishing (State 2) and electrolytic polishing (State 3). After Ar ion beam etching at 90° for 30 min, the diffraction pattern and the image quality are greatly enhanced (Fig. 2b). With prolonging the beam etching time, the image quality can be slightly improved (Fig. 2c). After ion beam etching at 80° for 30 min, the image quality is improved obviously (Fig. 2d). The image quality is further improved after ion beam etching at 70° for 30 min (Fig. 2e). The calibration result of Fig. 2e is shown in Fig. 2f. Compared with mechanical polishing and electrolytic polishing, the ion beam etching can achieve Be surface of higher quality.

The surface roughness R_a of Be specimen of State 2 was measured three times and the average value was used for analysis. As shown in Fig. 3, the average surface roughness of the original Be specimen of State 2 is 2.161 μm. The surface roughness is mainly affected by the etching duration, ion beam energy, current, and etching angle. R_a value becomes smaller and smaller with the etching proceeding. The surface roughness of 0.63 μm is achieved after Ar ion beam etching under the conditions of 600 eV, 100 mA, and 90° for 6 h.

2.2 Influence of specimen on measurement error

Partial Be specimen of State 1 was occluded by a mask, and the other part was etched by Ar^+ ion beam. After etching for 2 h (Fig. 4), no effective etching depth can be measured. This is because the etching rate on Be specimen is very slow, resulting in the measurement difficulty, even after etching for several hours. However, the mechanical scratches of several microns on the specimen surface can be observed. Besides, the etching depth cannot be accurately measured even on the polished surface of Be specimens of State 2, State 3, or State 4. The measurement errors of the Be specimens of State 1, State 2, State 3, or State 4 are unacceptable. In order to reduce the measurement error, the Be specimen with a thickness of dozens of microns (State 5) was used for etching.

2.3 Comparison of etching thickness measurement methods

Experimental etching rate (v) can be calculated by the ratio of etching thickness to etching time (t). The etching thickness is the difference between the specimen thickness before (h_1) and after (h_2) etching. Therefore, the thickness measurement is critical, and the etching rate can be calculated, as follows:

$$v=(h_1-h_2)/t \quad (1)$$

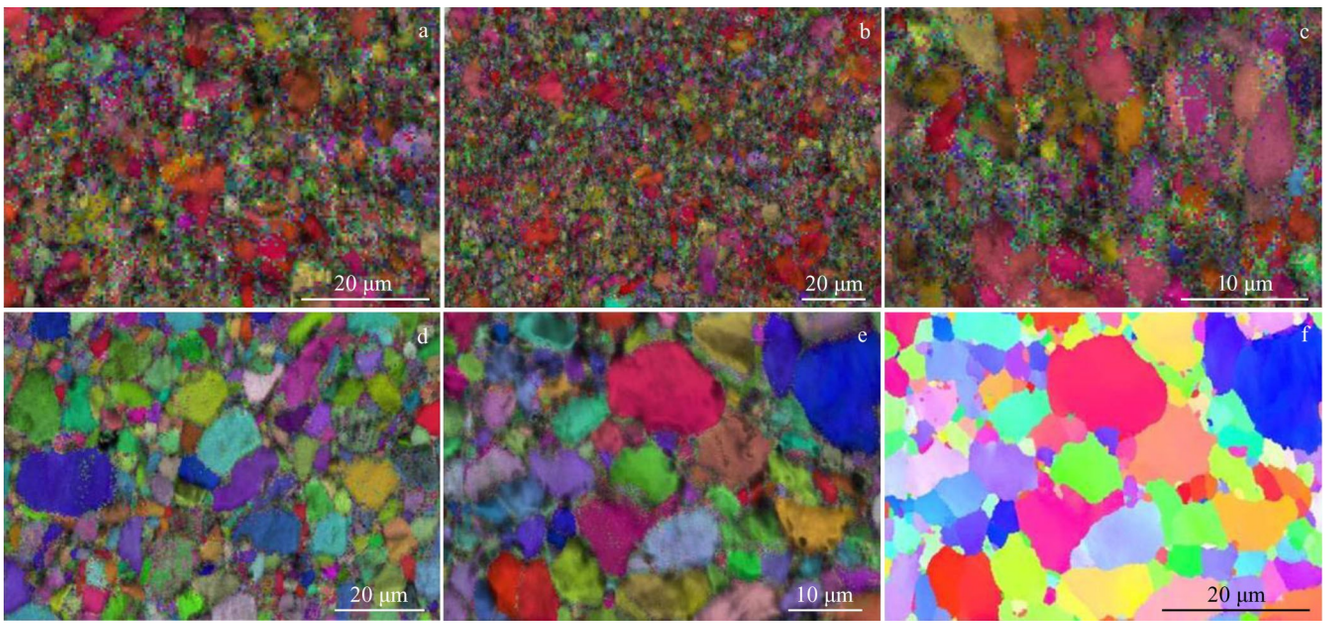

Fig.2 EBSD images of Be specimens after different steps of Ar ion beam etching treatment: (a) mechanical polishing and electrolytic polishing, (b) Ar ion beam etching at 90° for 30 min, (c) Ar ion beam etching at 90° for 90 min, (d) Ar ion beam etching at 80° for 30 min, and (e) Ar ion beam etching at 70° for 30 min; EBSD calibration result of Fig.2e (f)

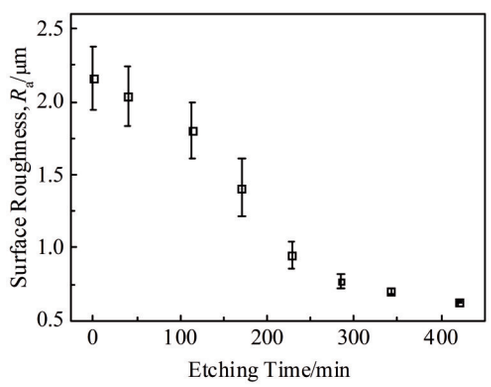

Fig.3 Surface roughness R_a of Be specimen of State 2 after Ar ion beam etching (600 eV, 100 mA) for different durations

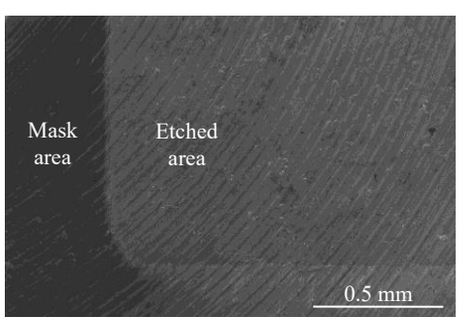

Fig.4 SEM morphology of Be specimen of State 1 after Ar ion beam etching for 2 h

2.3.1 WLI measurement

WLI was used to measure the thickness of Be specimen of State 5, and the results are shown in Fig.5. The Ar ion beam

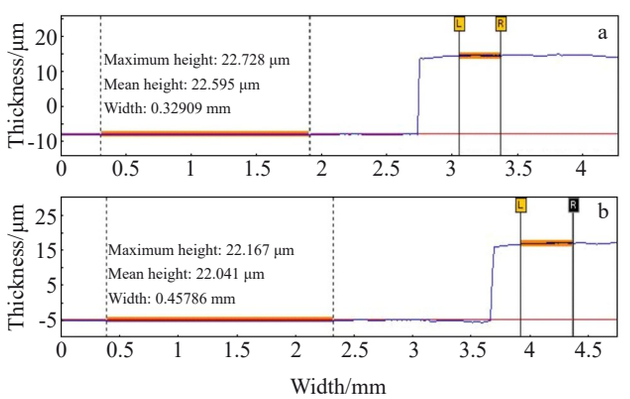

Fig.5 Thickness of Be specimen of State 5 before (a) and after (b) etching measured by WLI method

etching parameters are 600 eV, 60 mA, incident ion angle of 90°, and 60 min. The average thickness of Be specimen is 22.595 and 22.041 μm before and after etching, respectively. Thus, the calculated etching rate is 9.23 nm/min. The high measurement accuracy of WLI method is based on the flat specimen surface, which is suitable for optical observation. An inevitable system error exists in the process of measurement-etching-measurement. Therefore, WLI measurement method is not suitable for this research.

2.3.2 FIB measurement

Two Be specimens (Specimen 1 and Specimen 2) were fixed on a special tool. A hole of 15 μm×20 μm was cut by FIB method as the measurement point. For cross-section measurement, the placement stage of specimen was tilted at a certain angle. Then, the system automatically calculated the specimen thickness. The thickness of Specimen 1 and

Specimen 2 before etching is 27.38 (Fig. 6a) and 30.60 (Fig. 7a) μm, respectively. The relative positions of Specimen 1 and Specimen 2 on the tool did not change during transferring and etching processes. The etching was conducted at 700 eV, 95 mA, and incident ion angle of 90° for 80 min. As shown in Fig. 6b and Fig. 7b, after Ar ion beam etching, the edge steepness of Specimen 1 and Specimen 2 is decreased. Therefore, the magnified images of Fig. 6b and Fig. 7b were recorded for analysis, as shown in Fig. 6c and Fig. 7c. After etching, the thickness of Specimen 1 and Specimen 2 is 26.71 and 29.85 μm, respectively. Because FIB measurement method does not require contact, the system error is greatly reduced. The calculated etching rate of Specimen 1 and Specimen 2 is 10.47 and 11.66 nm/min, respectively. The average etching rate is 11.06 nm/min. Thus, FIB measurement is more suitable for the thickness measurement of Be specimens.

2.4 Effect of Ar ion beam energy on sputtering yield

The sputtering yield Y represents the average number of target atoms removed by an incident ion, which can be calculated through Eq.(2–4), as follows:

$$Y = \frac{w}{MIt} \times 10^5 \tag{2}$$

$$w=\upsilon tA\rho \tag{3}$$

$$I=JA \tag{4}$$

where w represents the material mass loss (g); M is the relative atomic mass of the target material; I is the incident ion beam (A); t is the etching time (s); A is the specimen area (cm^2); ρ is the density of etched material (g/cm^3); J is the beam density (A/cm^2).

According to Sigmund model[12], the sputtering yield as a function of sputtering energy E, $Y(E)$, can be calculated by Eq.(5), as follows:

$$Y(E)=0.042\alpha S(E)/E_s \tag{5}$$

where E_s is the sputtering threshold of the atoms on the target surface; α is a dimensionless quantity; $S(E)$ is the nuclear blocking cross section. The runaway atoms are regarded as the gaseous material. The sputtering threshold of the atoms can be replaced by the ascension heat U of material.

Bohdansky et al[13] proposed a novel formula based on Sigmund model, which considered both the electron stopping effect and the sputtering threshold, as follows:

$$Y(E) = \frac{0.042\alpha S(E)}{E_s}\frac{R_p}{R}\left[1-\left(\frac{E_s}{E}\right)^{2/3}\right]\left(1-\frac{E_s}{E}\right)^2 \tag{6}$$

where R_p/R is the ratio of average range R_p to projected range R. The Bohdansky empirical equation is more suitable for the calculation of light ion sputtering. R_p/R can also be replaced by Eq.(7), as follows:

$$R_p/R=1/(0.4M_2/M_1+1) \tag{7}$$

where M_1 and M_2 are the relative atomic mass of the incident ion and the target material, respectively. When the incident is vertical, α can be calculated by Eq.(8), as follows:

$$\alpha=0.15+0.13\ M_2/M_1 \tag{8}$$

In addition, ε represents the dimensionless amount of the lost energy; Z_1 and Z_2 are the atomic numbers of the incident ion and target material, respectively. Therefore, the parameter Z and ε can be expressed by Eq.(9) and Eq.(10), respectively:

$$Z = (Z_1^{2/3} + Z_2^{2/3})^{0.5} \tag{9}$$

$$\varepsilon = 0.032523M_2E/(M_1 + M_2)Z_1Z_2Z \tag{10}$$

The theoretical calculation of $S(\varepsilon)$ and $S(E)$ can be calculated by Eq.(11) and Eq.(12), as follows:

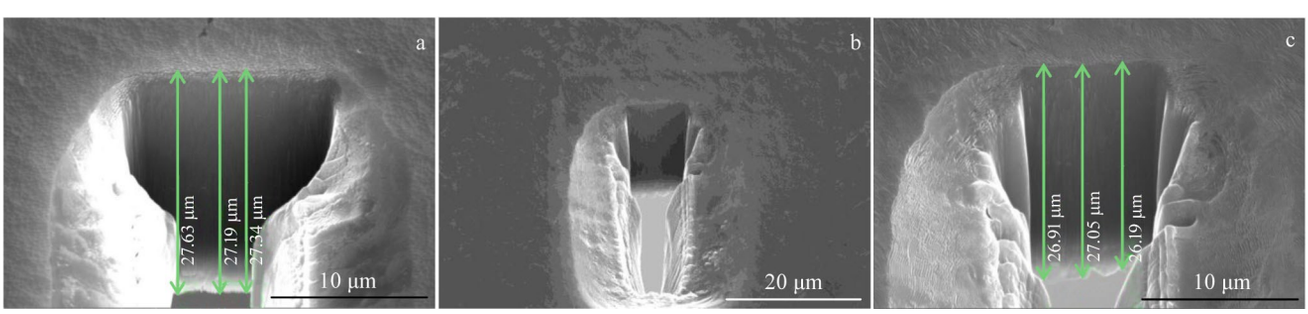

Fig.6 Appearances of Specimen 1 before (a) and after (b–c) etching

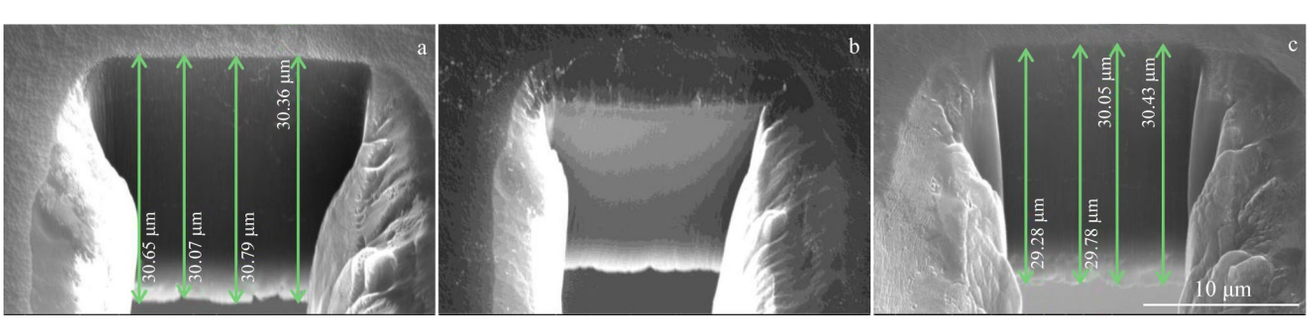

Fig.7 Appearances of Specimen 2 before (a) and after (b–c) etching

$$S(\varepsilon)=\frac{0.5\ln(1+1.2288\varepsilon)}{\varepsilon+0.0081\varepsilon^{0.15}+0.1728\varepsilon^{0.5}} \quad (11)$$

$$S(E)=\frac{84.445M_1Z_1Z_2S(\varepsilon)}{(M_1+M_2)Z} \quad (12)$$

According to Ref. [14], when ε is less than 0.1, the theoretical calculation of $S(\varepsilon)$ is in good agreement with the experiment result. In this research, ε is much less than 0.1, indicating the calculation accuracy.

The evaporation heat and melting heat of Be under standard conditions are 3.20 and 0.15 eV, respectively. Assuming that the ascension heat U of Be is 3.35 eV under standard conditions, the first ionization energy (U_{ion}) of Be is 9.32 eV.

According to Ref. [15], the sputtering yield calculated by Thomas-Fermi atomic interaction model coupled with Sigmund model is 0.4 after Ar sputtering of 0.4 keV on Be specimen, which is slightly higher than the experiment results in this research. In this research, the Thomas-Fermi atomic action potential and Bohdansky empirical equation were used. The sputtering yields are obtained under the sputtering thresholds of $1U$, $2U$, $3U$, $4U$, $5U$, ans U_{ion}, as shown in Fig.8. It can be seen that under the sputtering threshold of $3U$ and U_{ion}, the calculated sputtering yields are close to the experiment results in this research, suggesting that the sputtering process of Be by Ar ion bombardment is similar to the ionization process. The general equation of the sputtering yield of Ar ion on Be with the sputtering threshold of U_{ion} is shown in Eq.(13), as follows:

$$Y(E)=\frac{0.5783[1-(E_s/E)^{2/3}](1-E_s/E)^2\ln(1+1.4800\times10^{-4}E)}{1.2045\times10^{-4}E+2.0922\times10^{-3}E^{0.15}+1.8964\times10^{-3}E^{0.5}} \quad (13)$$

2.5 Effects of Ar ion beam current and sputtering yield on etching rate

According to the theoretical derivation, when the incidence is perpendicular to the Be surface, the relationship between the etching rate E_R of pure physical sputtering and beam density is as follows:

$$E_R=9.6\times10^{24}JY(E)/n \quad (14)$$

where J is the beam density (mA/cm^2), n is the atomic density of the etched material (atom/cm^3). The unit of E_R is nm/min. The atomic density of Be is 1.24×10^{23} atom/cm^3. Thus, the etching rate of Ar ion beam on Be can be calculated by Eq.(15), as follows:

$$E_R=77JY(E) \quad (15)$$

Under the experiment conditions, the etching rate calculated by Eq. (1) is compared with that calculated by Eq. (15), as shown in Fig. 9. The actual etching rate can be estimated through the theoretical calculation, and the variation of Be etching rate with the product of Ar ion beam energy and sputtering yield is obtained. The comparison results provide a guidance in the engineering application.

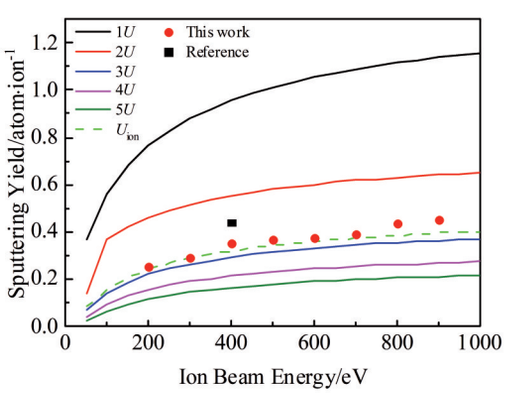

Fig.8 Sputtering yields under different Ar^+ energies and sputtering thresholds

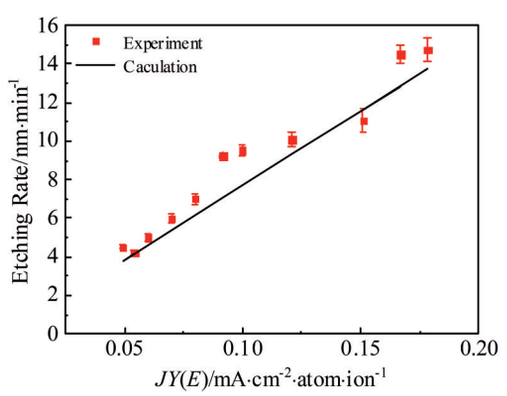

Fig.9 Comparison of experimental and calculated etching rates

3 Conclusions

1) Compared with mechanical polishing and electrolytic polishing, the Ar ion beam etching can achieve high-quality Be surface. With the Ar ion beam etching proceeding, the Be surface quality is improved gradually. The surface roughness of 0.63 μm is achieved after Ar ion beam etching for 6 h.

2) The focused ion beam (FIB) measurement method is more suitable for the thickness measurement of Be etching.

3) The Be sputtering process is similar to the ionization process of Be by Ar ion bombardment. The general equation of the sputtering yield of Ar ion on Be with the sputtering threshold of first ionization energy is $Y(E)=\frac{0.5783[1-(E_s/E)^{2/3}](1-E_s/E)^2\ln(1+1.4800\times10^{-4}E)}{1.2045\times10^{-4}E+2.0922\times10^{-3}E^{0.15}+1.8964\times10^{-3}E^{0.5}}$.

4) The etching rate of Ar ion beam on Be is related to the product of Ar ion beam energy and sputtering yield as $E_R=77JY(E)$.

5) This research provides guidance for the engineering application of Be etching.

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低能氩离子束对铍的刻蚀

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摘　要：研究了0~1000 eV的低能氩（Ar）离子束对Be的刻蚀。比较了多种不同的表面抛光方法，结果发现Ar离子束刻蚀能获得高质量的Be表面。随着刻蚀的持续进行，Be表面质量逐渐提升，表面粗糙度在600 eV和100 mA的条件下刻蚀6 h后趋于稳定，达到了0.63 μm。比较了白光干涉（WLI）和聚焦离子束（FIB）的测量方法，发现FIB测量法更适合Be刻蚀深度的测量。实验结果和理论计算表明，Be的溅射过程与其被Ar离子轰击后的电离过程较为接近。采用第一电离能作为溅射阈值，获得了Ar离子能量对Be溅射产额的影响规律，获得了Be刻蚀速率随Ar离子束流和溅射产额乘积的变化规律，为Be刻蚀的工程应用奠定了基础。

关键词：氩离子束；铍；刻蚀

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