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Towards Atomic-scale Smooth β-Ga_2O_3 Surfaces Manufacturing via Atmospheric Pressure Inductively Coupled Plasma

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β-Ga_2O_3 is an emerging next-generation ultra-wide bandgap semiconductor (UWBG) that shows great application potentials in novel high-power, high-frequency, and high-temperature semiconductor devices. However, β-Ga_2O_3 is indeed a typical difficult-to-machine material owing to its strong mechanical strength and chemical stability. Herein, we proposed an atomic-scale smoothing strategy, including two modes, namely plasma etching polishing (PEP) and plasma-induced atom migration manufacturing (PAMM), for β-Ga_2O_3 (010) substrate via atmospheric pressure inductively coupled plasma (ICP). We found that although the high-input-power CF_4-O_2-Ar plasma can achieve the polishing of β-Ga_2O_3, the co-existence of etching and migration effects would deteriorate the surface and prevent the further reduction of the surface roughness. We then adopted the low-input-power CF_4-O_2-Ar plasma and the high-input-power Ar plasma to treat the β-Ga_2O_3 substrate, and found that the scratched β-Ga_2O_3 can be polished to the atomic-scale smooth level with the Sa surface roughness of 0.424 nm and 0.347 nm mainly through the etching and migration processes, respectively. The PEP and PAMM methods proposed here are highly efficient, compared with the widely used chemical mechanical polishing (CMP). We believe that both PEP and PAMM would greatly promote the further development of β-Ga_2O_3-based power electronics.

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1. Introduction

β-gallium oxide (β-Ga_2O_3) is an emerging next-generation semiconductor with high melting point (1900 ℃), large bandgap (4.85 eV), large electron saturation velocity (above 1.2×10^7 cm/s), and high breakdown field (8 MV/cm), which makes it a promising candidate for certain classes of novel power electronics that can work at high-temperature, high-power, and high-frequency occasions, such as solar-blind ultraviolet photodetectors, metal-semiconductor field effect transistors (MESFETs), and Schottky barrier diodes (SBDs)[1-3]. To maximize the performance of β-Ga_2O_3-based power electronics, a damage-free and atomic-scale smooth surface is generally necessary, which is a significant field of atomic and close-to-atomic scale manufacturing (ACSM)[4]. However, β-Ga_2O_3 is a typical difficult-to-machine material as it shows strong mechanical strength and chemical stability[1,5,6]. Currently, there are mainly two approaches to achieve the atomically smooth β-Ga_2O_3 surfaces manufacturing. One is widely known as chemical mechanical polishing (CMP) and the other is high-temperature annealing. Huang et al. showed that the as-received rough β-Ga_2O_3 crystal substrate with numerous scratches and pits found on the surface can be polished to the damage-free and smooth surface with the Rq surface roughness of 0.21 nm when using silica sol as slurry during the CMP process[5]. Xu et al. also demonstrated that the root-mean-square (RMS) surface roughness of β-Ga_2O_3 thin film deposited on the SiC substrate can be reduced from 5.43 nm to 0.8 nm after CMP process[7]. On the other hand, Ohira et al. proposed that an atomically smooth β-Ga_2O_3 surface with the uniform step-terrace structures can be obtained after the annealing at 1100 ℃ for 3 h[8]. Although both CMP and high-temperature annealing can be utilized to achieve the atomic-scale smooth β-Ga_2O_3 surface manufacturing, these two approaches generally consume several hours, ultimately leading to their quite low efficiency. Thus, exploring highly-efficient atomic-scale smooth surface manufacturing technique for β-Ga_2O_3 is of great importance to promote its applications in power electronics.

Herein, we proposed an atomic-scale smoothing strategy for β-Ga_2O_3 (010) substrate via atmospheric pressure inductively coupled plasma (ICP), in which two completely different polishing modes, namely plasma etching polishing (PEP) and plasma-induced atom migration manufacturing (PAMM), were fully discussed. We found that high-input-power CF_4-O_2-Ar plasma can successfully polish the rough β-Ga_2O_3 (010) surface with apparent scratches to a smooth surface with the *S*a surface roughness of 1.45 nm, mainly through the PEP process in the relatively short plasma-treated duration time; however, once the treatment time exceeded 3 min, the migration effect would play a huge role and the co-existence of etching and migration effects would severely deteriorate the surface. Thus, to separate the etching and migration effects, we adopted low-input-power CF_4-O_2-Ar plasma and high-input-power Ar plasma to treat the as-received rough β-Ga_2O_3 (010) surface, respectively. It was found that the *S*a surface roughness of β-Ga_2O_3 was reduced to 0.424 nm after the around two-minute etching of CF_4-O_2-Ar plasma at 600 W and the Ar plasma (at 900 W)-induced migration effect could lead to the *S*a surface roughness of 0.347 nm within about 40 minutes. We believe that the two plasma-based polishing methods proposed here can provide new sights into the ACSM for β-Ga_2O_3 and would promote its further applications in various electronics.

2. Plasma setup and diagnostics

2.1 Plasma setup

Fig. 1 shows the atmospheric pressure ICP setup used in this study. It is apparent from the schematic diagram in Fig. 1(a) that this plasma setup mainly consists of matcher, radio frequency (RF) power, electric sparker, double concentric quartz torch, and water cooler. Once the cooling gas (Ar), ignition gases (Ar), and/or reaction gases (CF_4/O_2) are supplied into this system, plasma can be generated at the outlet of the quartz torch and the β-Ga_2O_3 (010) substrate placed on a sample holder in the 3-axis numerical control (NC) platform would be irradiated by the plasma. Figs. 1(b) and 1(c) show the photos of CF_4-O_2-Ar plasma and Ar plasma, respectively, where CF_4 and O_2 are added as reaction gases for the former and only Ar is used for the latter. The differences between these two types of ICP can be primarily seen from their colors. Obviously, the color would change from the bright purple to the light green if the reaction gases (CF_4 and O_2) are introduced into the Ar plasma. More details about the different properties of these two ICP will be discussed in the section of plasma diagnostics.

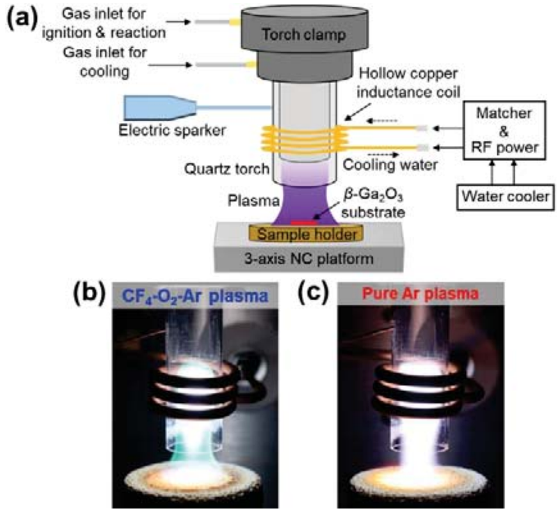

Fig. 1 ICP setup. (a) Schematic diagram of atmospheric pressure ICP system. Photos of (b) CF_4-O_2-Ar plasma and (c) pure Ar plasma.

2.2 Plasma diagnostics

Atmospheric pressure ICP is generally believed to present high-temperature and high-radical-density characteristics[9,10]. Here, we used an infraction imager (FLIR 600) to quantitatively measure the variation of β-Ga_2O_3 surface temperature with the input power of ICP changing from 600 W to 1200 W and an optical emission spectroscopy (OES, Ocean Optics USB4000) to qualitatively explore the radical particles in ICP. Fig. 2(a) shows the temperature measurement results for both Ar plasma and CF_4-O_2-Ar plasma, where the flows for CF_4, O_2, Ar for ignition, and Ar for cooling are 60 sccm, 20 sccm, 1.5 slm, and 18 slm, and the substrate-to-torch distance is around 12 mm. Clearly, the surface temperature of β-Ga_2O_3 substrate increases with the input power whether treated by Ar plasma or CF_4-O_2-Ar plasma. However, it is worth mentioning that at the same input powers, the surface temperatures (T_1) when using Ar plasma are slightly higher than that (T_2) when using CF_4-O_2-Ar plasma, for example, T_1 = 1081 ℃ and T_2 = 1040 ℃ at 900 W. In addition, based on the OES results shown in Fig. 2(b), it can be found that many extra radicals (C, C_2, and CF_x) are detected in CF_4-O_2-Ar plasma when compared with Ar plasma, which indicates that the dissociation of CF_4 can occur once being added into the plasma as one of the reaction gases. Note that O_2 gas in CF_4-O_2-Ar plasma is utilized to promote the dissociation of CF_4 and the OES peaks corresponding to F and O radicals are not shown in Fig. 2(b) due to the overlap of the strong Ar peaks. Later, we will show that both the high-temperature and high-radical-density features of ICP can be used to achieve the atomic-scale smooth surface manufacturing for β-Ga_2O_3 (010) substrate.

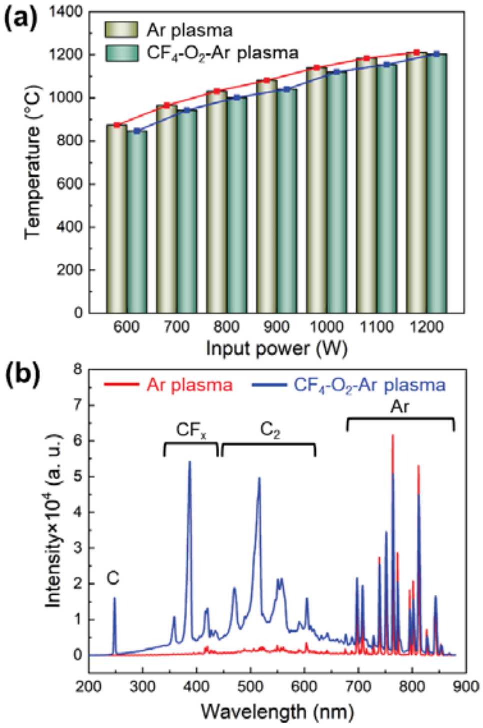

Fig. 2 Plasma diagnostics. (a) The variation of β-Ga_2O_3 (010) surface temperature with the increase of input power from 600 W to 1200 W. (b) Typical OES results of Ar plasma and CF_4-O_2-Ar plasma.

3. Results and discussion

3.1 High-input-power CF_4-O_2-Ar plasma treating of β-Ga_2O_3

The fluorine radicals in CF_4-O_2-Ar plasma possess strong oxidation potential, by which the β-Ga_2O_3 substrate can be etched through the following reaction,

$$CF_4 + Ga_2O_3 \rightarrow GaF_3 + CO/CO_2.$$

Figs. 3(a)-3(f) show the processing results using high-input-power (900 W) CF_4-O_2-Ar plasma, which are obtained by an atomic force microscope (AFM, BRUKER Dimension Edge in tapping mode). It is easy to find that the as-received β-Ga_2O_3 (010) surface (Fig. 3(a)) with many scratches can be rapidly etched by CF_4-O_2-Ar plasma within only 30 s and this short-time etching effect would greatly roughen the surface with the emergency of peak-valley structures (Fig. 3(b)). After one-minute treatment, many hemispherical etching pits can be observed on the surface (Fig. 3(c)). The extension of duration time to 3 min leads to an atomic-scale smooth surface with the *S*a surface roughness of only 1.45 nm, on which no scratches can be found (Fig. 3(d)). However, further prolonging the duration time would deteriorate the surface roughness, as shown in Figs. 3(e) and 3(f).

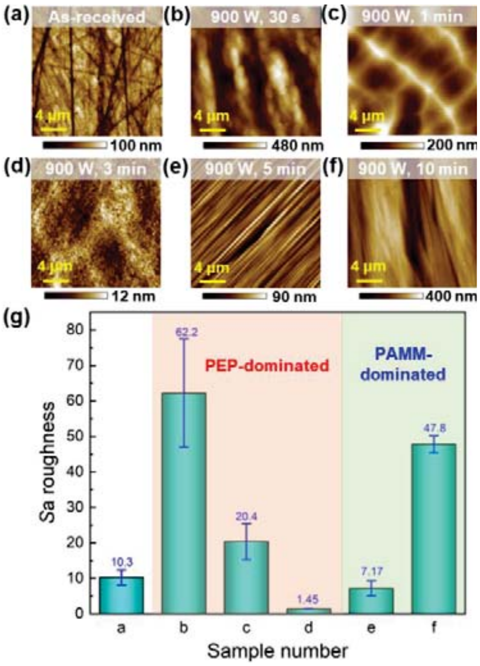

Fig. 3 CF_4-O_2-Ar plasma processing results at 900 W. (a)-(f) AFM images of β-Ga_2O_3 (010) surfaces with different plasma-treated duration times. (g) *S*a surface roughness of the surfaces in (a)-(f).

According to the temperature measurement results in Fig. 2(a), the β-Ga_2O_3 surface temperature should be around 1081 ℃ when the input power of CF_4-O_2-Ar plasma is 900 W; under this condition, the high-temperature-induced migration effect on surface atoms would play a great role during the plasma treatment process. In our recent studies, we find that it is the co-existence of etching and migration effects that consequently deteriorates the surface. Fig. 3(g) shows the variation of *S*a surface roughness for the different plasma-treated duration times, and the whole plasma treatment process can be roughly classified into two stages, namely PEP-dominated stage and PAMM-dominated stage. In detail, the scratched substrate can be speedily polished mainly through the etching process in the PEP-dominated stage; once the smooth surface is formed, the migration effect would be magnified; in the subsequent PAMM-dominated stage, the co-promotion of etching and migration effects makes the surface rough again. To obtain the atomically smooth β-Ga_2O_3 (010) surface, we next try to separate the etching and migration effects by using low-input-power CF_4-O_2-Ar plasma (mainly etching effect) and high-input-power Ar plasma (only migration effect).

3.2 PEP of β-Ga_2O_3 (010) substrate

Fig. 4 shows the PEP results of β-Ga_2O_3 (010) substrate using CF_4-O_2-Ar plasma at 600 W, under which the surface temperature is approximately 874 ℃ (Fig. 2(a)) where the migration effect should be greatly depressed. Here, the sample was treated sequentially for 10 s, 30 s, and 60 s. According to the images obtained by a laser scanning confocal microscopy (LSCM, KEYENCE VK-X1000) in Figs. 3(a)-3(c), the rough β-Ga_2O_3 (010) surface can be rapidly polished to the atomic-scale level and all scratches are totally removed only after the sixty-second etching. The corresponding AFM images in Figs. 4(d)-4(f) indicate that the surface becomes smoother and smoother with the increase of duration time and the final *S*a surface roughness is reduced to 0.424 nm, which is much smaller than that shown in Fig. 3(d). The above results demonstrate that by decreasing the input power of CF_4-O_2-Ar plasma from 900 W to 600 W, the migration effect induced by the high temperature is definitely depressed to a great extent. Thus, a smoother β-Ga_2O_3 (010) surface is consequently obtained in a shorter plasma-etching duration time.

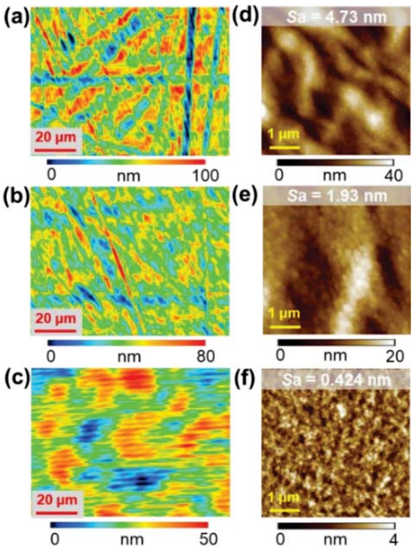

Fig. 4 PEP results of β-Ga_2O_3 (010) substrate at 600 W. (a)-(c) LSCM images of the surfaces treated for 10 s, 30 s, and 60 s. respectively. (d)-(f) Corresponding AFM images of (a)-(c).

3.3 PAMM of β-Ga_2O_3 (010) substrate

Fig. 5 shows the PAMM results of β-Ga_2O_3 (010) substrate using Ar plasma at 900 W, under which the etching effect originating from the fluorine radicals is completely removed and the surface temperature is evaluated to be around 1040 ℃ (Fig. 2(a)). Studies have revealed the migration effect of surface atoms on β-Ga_2O_3 substrate at high temperature conditions[8]. Herein, by sequentially treating the scratched β-Ga_2O_3 (010) substrate for 1 min, 3 min, 5 min, 10 min, 15 min, and 20 min, we find that the high-input-power (high-temperature) Ar plasma can also promote the surface atoms' migration on the β-Ga_2O_3 (010) substrate, ultimately resulting in an atomic-scale smooth surface. Specifically, the LSCM images in Figs. 5(a)-5(c) present the migration results on a large scale (note that the results for 3 min, 5 min, and 15 min are not shown here), where it can be found that the scratches are gradually recovered. After the twenty-minute treatment, no scratches can be found on the surface. The AFM images in Figs. 5(d)-5(f) indicate that the high-temperature induced migration effect can also lead to the smoothing of β-Ga_2O_3 (010) substrate and the ultimate *S*a surface roughness is 0.347 nm, which is smaller than that in Figs. 3(d) and 4(f). But the efficiency should be much lower than that of the low-input-power PEP process. We further infer that the smoothing process in PAMM may follow the rule of surface energy reduction.

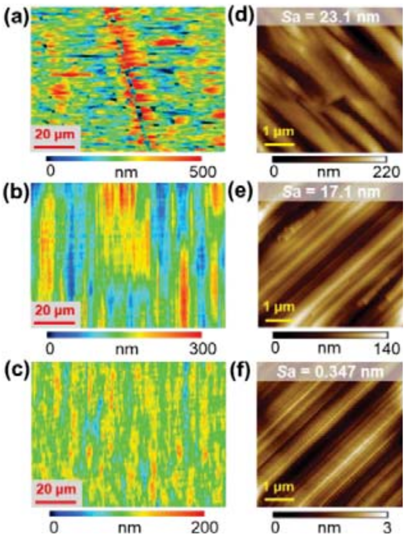

Fig. 5 PAMM results of β-Ga_2O_3 (010) substrate at 900 W. (a)-(c) LSCM images of the surfaces treated for 1 min, 10 min, and 20 min, respectively. (d)-(f) Corresponding AFM images of (a)-(c).

4. Conclusions

In this study, we proposed two fairly efficient and damage-free polishing modes, namely PEP and PAMM, to achieve the atomic-scale smooth β-Ga_2O_3 (010) surface manufacturing based on atmospheric pressure ICP. To sum up, the following conclusions can be drawn.

(1) The high-input-power (900 W) CF_4-O_2-Ar plasma can polish the scratched and rough β-Ga_2O_3 (010) surface to a smooth surface with the *S*a surface roughness decreasing from 10.3 nm to 1.45 nm. Further decrease of surface roughness is restricted due to the co-existence of etching and migration effects.

(2) Adopting the low-input-power (600 W) CF_4-O_2-Ar plasma, in which the high-temperature-induced migration effect would be greatly weakened, can rapidly smoothen the β-Ga_2O_3 (010) substrate to the atomic-scale level, and the final *S*a surface roughness is only 0.424 nm after about two-minute etching. This PEP mode is obviously of high efficiency when compared with the conventional CMP and high-temperature annealing methods.

(3) The high-input-power (900 W) Ar plasma, in which only the high-temperature-induced migration effect exists, can also polish the β-Ga_2O_3 (010) surface to the atomic-scale smooth state with the *S*a surface roughness of 0.347 nm, following the rule of surface energy reduction. Although the PAMM mode is not as efficient as PEP, the efficiency of PAMM is still much higher than that of CMP and high-temperature annealing.

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