euspen's 16th International Conference & Exhibition, Nottingham, UK, May 2016
www.euspen.eu

Electrochemical mechanical polishing of difficult-to-machine mold materials

Kazuya Yamamura [1], Yusuke Imanishi [1], Katsuyoshi Endo [1]

[1] *Research Center for Ultra-precision Science and Technology, Graduate School of Engineering, Osaka University, Japan*

Email: yamamura@upst.eng.osaka-u.ac.jp

Abstract
CVD-SiC, reaction-sintered SiC (RS-SiC), tungsten carbide (WC) and glassy carbon (GC) are suitable materials for high-precision glass forming mold because of their excellent properties. Chemical mechanical polishing (CMP) is generally applied to finish the surface of those materials. However, polishing rates of those materials are very low due to the high-hardness and chemical inertness of mold materials. Furthermore, cost of CMP is relatively high because both purchase and waste disposal costs of slurry are expensive, and strict control of slurry condition is essential to maintain the polishing properties constant. To resolve these issues, we proposed application of slurryless electrochemical mechanical polishing (ECMP) using fixed abrasive grain to finishing of difficult-to-machine materials. SiC, WC and GC are anodically oxidized in electrolyte by applying positive potential to the substrate because these materials have electrical conductivities. Hardness of the anodically oxidized surface drastically decreases and this softening enables us to increase the polishing rate of the difficult-to-machine material. We applied a resin bonded grinding stone containing silica or ceria abrasive to polish the oxidized surface. Since the hardnesses of both silica and ceria abrasive are softer than that of mold material, scratch free and subsurface damage (SSD) free surface is obtained by applying ECMP. In this study, we demonstrated preliminary experimental results on slurryless ECMP of monocrystalline SiC substrate using resin bonded ceria abrasive grinding stone, and rms roughness of 0.12 nm was obtained by applying low anodic oxidation potential in which removal rate of oxide was higher than anodic oxidation rate.

Glass forming mold, Slurryless, Polishing, Difficult-to-machine material, Anodic oxidation, Electrochemical mechanical polishing

1. Introduction

Difficult-to-machine materials such as CVD-SiC, reaction-sintered SiC (RS-SiC), tungsten carbide (WC) and glassy carbon (GC) are considered as suitable materials for high-precision glass forming mold because of their excellent properties such as high-hardness, durability against high-temperature oxidation, chemical inertness and so forth. To finish the surface of those materials, chemical mechanical polishing (CMP) is generally applied. However, polishing rates of those materials in CMP are very low due to the high-hardness and chemical inertness of mold materials. Furthermore, cost of CMP is relatively high because both purchase and waste disposal costs of slurry are expensive, and in the case of semiconductor wafer manufacturing process, many consumables such as polishing pad, packing material, dressing material, conditioning material, and cleaning solution are needed. Furthermore, strict control of slurry condition by dispersion of abrasive and filtration of slurry is essential to maintain the polishing properties constant, and to avoid formation of scratches on the surface. Those requirements need additional equipment to the main system, and it also results in increase in initial cost. To resolve these issues, we proposed two hybrid finishing processes which combined surface modification process and mechanical removal process of modified layer utilizing soft abrasive compared to the base material. One process is dry finishing technique named plasma assisted polishing (PAP) which combined surface modification by atmospheric pressure plasma irradiation and mechanical polishing utilizing resin bonded soft abrasive [1]. By applying PAP, atomically smooth surface without subsurface damage (SSD) and etch pit for single crystal 4H-SiC (0001) and GaN (0001) [2-4]. PAP is also useful for finishing of CVD-SiC surface in the sense of removal efficiency and low surface roughness [5]. The other one technique is electrochemical mechanical polishing (ECMP) which combined surface modification by anodic oxidation and removal of modified layer by soft abrasive. Hardness of the anodically oxidized surface drastically decreases and this softening enables us to increase the polishing rate of the difficult-to-machine material. We have already applied ECMP to finishing of RS-SiC and 4H-SiC (0001) [5-9]. In our previous results show that ECMP using ceria slurry as an electrolyte is promising technique for highly efficient removal of subsurface damage since anodic oxidation rate is relatively high compared to the plasma oxidation and thermal oxidation, and damage region is preferentially oxidized [9]. In this study, we newly propose the slurryless ECMP technique using resin bonded grinding stone. Material removal rate (MRR) and surface roughness in ECMP strongly depend on the balance between the oxidation rate and oxide removal rate [8]. In our proposed slurryless ECMP system, oxidation rate and oxide removal rate can be controlled independently because electrical conductivity and pH of the electrolyte and material of the abrasive can be chosen freely. In this paper, we describe preliminary experimental results on slurryless ECMP of monocrystalline SiC substrate using resin bonded ceria abrasive grinding stone.

2. Experimental setup

Fig. 1 shows the experimental setup of slurryless ECMP used for our preliminary research. Workpiece was set on the bottom of the container filled with an electrolyte and was connected to the working electrode (WE) of the potentiostat from the backside of the workpiece. Rotatable polishing head was constructed from the grinding stone part and electrode part

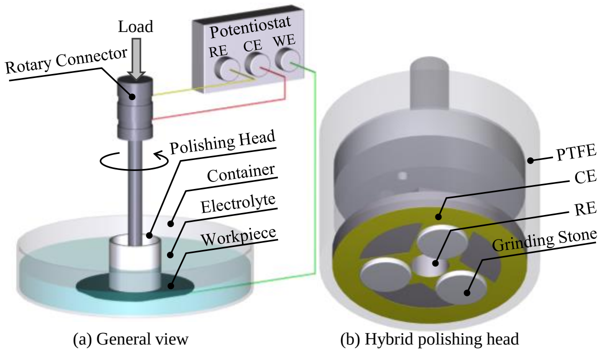


Figure 1. The experimental setups used for slurryless ECMP.

connected to the counter electrode (CE) of the potentiostat. Reference electrode (RE) of the potentiostat was installed at the center of the polishing head. Grinding stone was composed by resin bonded ceria abrasive with a mean diameter of 0.7 µm. Diameter of the polishing head and resin bonded grinding stone, and center position of the grinding stone from the center of the polishing head were 34 mm and 8 mm, and 7 mm, respectively. Polishing pressure was loaded by dead weight. To evaluate polishing properties of slurryless ECMP simply, CMP finished 2 inch sized n-type 4H-SiC (0001) wafer with specific resistance of < 0.1 Ωcm was used as a specimen. Electrolyte was NaCl (1 wt%, 1.8 S/m), and potentials (WE) of specimen to the RE (Ag/AgCl) were 3 V and 5 V. Polishing pressure and rotation speed of polishing head were 16 kPa and 500 rpm, respectively.

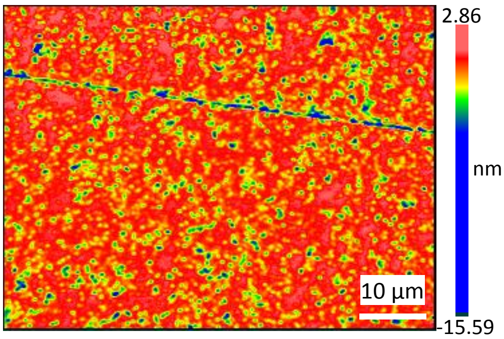
3. Results and discussion

Fig. 2(a)(b)(c) show the processed 4H-SiC (0001) surface measured by scanning white light interferometer (SWLI). In the case of applying anodic oxidation without polishing, surface roughness increased and scratch like structure emerged on the surface as shown in Fig. 2(a). Since there was no scratch on the CMP finished surface before anodic oxidation, it was assumed that subsurface damage (SSD) introduced before CMP was the cause of scratch formation.

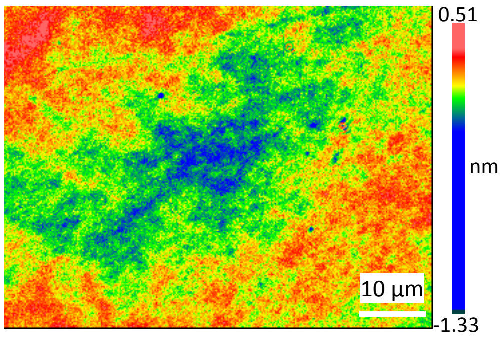
In the case of ECMP with low anode potential (3 V), scratch free smooth surface with an rms roughness of 0.12 nm was obtained as shown in Fig. 2 (b). This roughness value is almost the same with the roughness of SiC wafer finished by conventional CMP even though using relatively large size abrasive (0.7 µm). It is assumed that oxidation process is the main factor to decrease the surface roughness of SiC rather than the size of abrasive, since roughness of the interface between oxide and base material (SiC) is relatively smooth while the thickness of the oxide is thin [2].

In contrast, in the case of high anode potential (5 V), scratch was observed on the surface, and the surface roughness increased as shown in Fig. 2 (c). In our previous results showed that anodic oxidation rate of SiC is relatively high compared with the plasma oxidation and thermal oxidation, and the roughness of oxide/SiC interface tended to increase with the increase in thickness of the oxide [9]. Removal rate of oxide by resin bonded grinding stone was constant in both ECMP experiment with anodic potential of 3 V and 5 V. From above reasons, it is assumed that oxidation rate in the high anodic potential condition, which was higher than the removal rate of oxide, caused increase in surface roughness of SiC substrate.

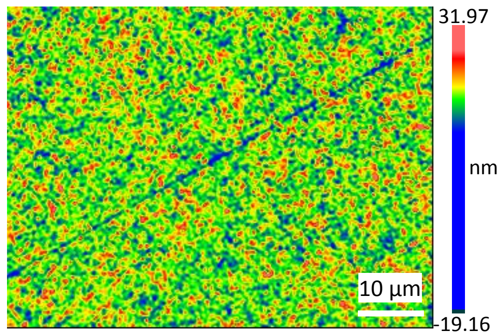
These experimental results lead to the conclusion that high removal rate of the oxide superior than the anodic oxidation rate is essential condition to obtain an atomically smooth surface in ECMP finishing.



(a) Anodic oxidation w/o polishing (rms 0.955 nm).



(b) ECMP with WE of 3 V (rms 0.116 nm).



(c) ECMP with WE of 5 V (rms 4.790 nm).

Figure 2. Surface morphologies of processed SiC substrate.

4. Conclusions

Preliminary research on slurryless ECMP of 4H-SiC (0001) substrate using NaCl electrolyte and resin bonded ceria abrasive grinding stone was conducted. Experimental results showed that rms roughness of 0.12 nm was obtained by applying low anodic potential (3 V) but increased by applying high anodic potential (5 V). From these results, it is concluded that balance between the anodic oxidation rate and the oxide removal rate is very important determining factor to obtain smooth surface in ECMP process.

5. Acknowledgments

This work was partially supported by a research and development grant from the Mazak foundation. We also acknowledge the Noritake Co., Ltd. for donating the resin bonded ceria grinding stone.

References

[1] Yamamura K, *et al.* 2011 *Annals of the CIRP* **60** 571-574
[2] Deng H, *et al.* 2013 *Annals of the CIRP* **62** 575-578
[3] Deng H, *et al.* 2014 *Annals of the CIRP* **63** 529-532
[4] Deng H, *et al.* 2015 *Annals of the CIRP* **64** 531-534
[5] Deng H, *et al.* 2015 *Proc. of euspen* 359-360
[6] Shen X, *et al.* 2013 *Opt. Express* **21** 14780-14788
[7] Shen X, *et al.* 2013 *Opt. Express* **21** 26123-26135
[8] Yamamura K, *et al.* 2014 *Adv. Mat. Res.* **1017** 509-514
[9] Deng H, *et al.* 2015 *Electrochem. Commun.* **52** 5-8