PAPER • OPEN ACCESS

Investigation on machinability of the oxide layer in anodic oxidation of reaction-sintered silicon carbide by pure-water

View the article online for updates and enhancements.

IOP ebooks™

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

Investigation on machinability of the oxide layer in anodic oxidation of reaction-sintered silicon carbide by pure-water

Xiaocui Yang[1], Xinmin Shen[1, 2, *], Zhizhong Li[1], Qunzhang Tu[1], and Qin Yin[1]

[1]College of Field Engineering, Army Engineering University, No. 1 Haifu Street, Nanjing, Jiangsu, P. R. China

[2]Department of Industrial and Systems Engineering, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong SAR, P. R. China

Email: shenxmjfjlgdx2014@163.com; x.m.shen@polyu.edu.hk

Abstract. Oxidation-assisted polishing has been developed as an efficient method for precision machining of reaction-sintered silicon carbide (RS-SiC), and characteristic of the oxide layer is the critical factor to obtain a fine surface property. Machinability of the oxide layer obtained in anodic oxidation of RS-SiC by the pure-water under high-frequency-square-wave potential is investigated by the ceria slurry polishing in this study. Quantitative analysis of surface quality of the oxidized RS-SiC sample is conducted by the scanning white light interferometer (SWLI) measurement. Along with increase of the oxidation time, surface quality of the oxidized RS-SiC is changing better at the beginning, and rapidly deteriorated in further oxidation process. Surface qualities of the RS-SiC sample before oxidation, after oxidation, after HF etching, and after abrasive polishing, are compared by the SWLI measurement. Surface roughness rms after anodic oxidation for 60min is 189.004nm. After removing the oxide by ceria slurry polishing, the rms can reach 3.688nm. Meanwhile, there is no visible scratch on the new revealed surface. Therefore, combination of anodic oxidation of RS-SiC by pure water and abrasive polishing of oxide layer by ceria slurry can be considered as an efficient method to machine RS-SiC.

1. Introduction

Oxidation-assisted polishing is a novel developed method for precision machining of reaction-sintered silicon carbide (RS-SiC), which is aim to promote the material removal rate (MRR) in figuring process and improve the surface quality in finishing process [1-3]. Relative to the ultraviolet Irradiation [4, 5], thermal oxidation [6-8], or plasma oxidation [9, 10], the anodic oxidation has the advantages of a high oxidation rate [11-14], which makes it a focus of research in the machining of RS-SiC.

Several electrolytes have been used to anodically oxidize RS-SiC, such as phosphoric acid (H_3PO_4) [11], hydrogen peroxide (H_2O_2) accompanied with hydrochloric acid (HCl) [13], and so on. However, fabrication of these electrolytes and post-process of these abandoned electrolytes have pollution to the environment [15]. Thus, anodic oxidation of RS-SiC by pure water only under high-frequency-square-wave potential is conducted, and machinability of oxide layer is investigated by ceria slurry polishing in this research. Quantitative analysis of surface quality of the oxidized RS-SiC is conducted by the scanning white light interferometer (SWLI) detection [16-18]. Meanwhile, surface qualities of RS-SiC before oxidation, after oxidation, after HF etching, and after abrasive polishing, are compared. By analysis of surface quality and validation of machinability of the oxide layer, the feasibility to machine RS-SiC through anodic oxidation by pure water with assistance of abrasive polishing are conducted.

2. Experimental design

The abrasive polishing system shown in figure 1 is used to validate machinability of the oxide in the anodic oxidation of RS-SiC. The abrasive is ceria (CeO_2) slurry. The oxidized sample is fixed on the stage, and surface of the oxidized sample contacts the ceria slurry. The polishing pad is immersed into the ceria slurry, and surface of the oxidized RS-SiC sample is abraded by the polishing pad. The load can be adjusted by adjusting the weights on the load plate. The oxidized RS-SiC sample, which was oxidized for 60min, is polished by the abrasive polishing system. Parameters in the abrasive polishing process are summarized in Table 1. Afterwards, the polished sample is detected by SEM observation to investigate the surface morphologies and by SWLI measurement to evaluate the surface qualities.
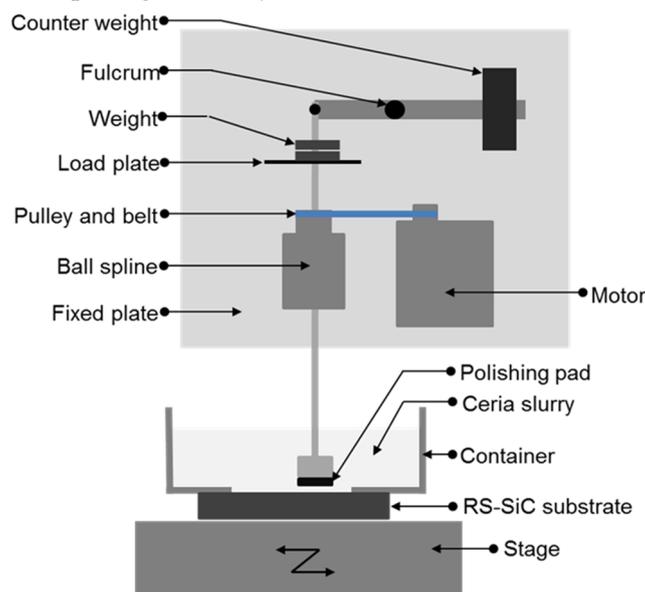

Figure 1. Schematic diagram of the abrasive polishing system.

Table 1. Parameters in the abrasive polishing process.

Parameters	Values
Sample and its dimension	Oxidised RS-SiC, 15mm×15mm
Abrasive type and its dimension	Ceria, ϕ190nm
Concentration of the slurry	0.1wt%
Downward pressure and load	12.475kPa, 100g
Scanning speed and rotation speed	100mm/min, 300rpm
Polishing pad and its dimension	K0017 (FILWEL Co. Ltd.), ϕ10mm

3. Results and discussions

For the purpose of quantitative analysis of the oxide morphologies, the initial surface obtained by the diamond lapping, the oxidized surface when the oxidation time is 10min, and that when the oxidation time is 60min are investigated by SWLI measurement, respectively. The results are summarized in the figure 2. Since hardness of RS-SiC is smaller than that of diamond, there are scratches on the original surface, as shown in figure 2(a). Surface roughness rms of the original RS-SiC sample in figure 2(a) is 2.814nm. After oxidation for 10min, the surface roughness rms is improved to 2.144nm, as shown in figure 2(b). The reason for this phenomenon is that the oxidation of RS-SiC is a volume expansion process. Therefore, the expanded oxides can fill the scratches which are generated from the diamond lapping of RS-SiC. Meanwhile, the oxidation time is short, so the projections and cracks generated by the volume expansion force are not obvious. However, when the oxidation time reaches 60min, the surface roughness rms is rapidly deteriorated to 189.004nm, as shown in figure 2(c). The reason for this phenomenon is that sizes of the projects and cracks are gradually increasing.
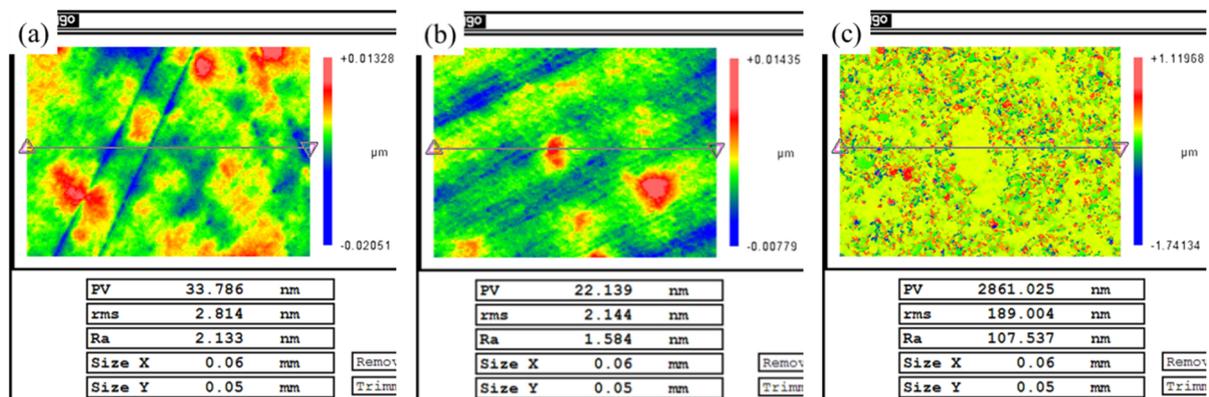
Figure 2. Quantitatively analysis of surface morphologies of the oxidized RS-SiC sample by SWLI measurement. (a) the initial RS-SiC surface. (b) oxidation for 10min. (c) oxidation for 60min.

After oxidation for 60min, the oxidized RS-SiC sample is polished by the ceria slurry polishing system shown in figure 1. When the oxide is completely removed, the RS-SiC sample is detected by SEM observation, and the cross-sectional surface morphologies are shown in the figure 3. Schematic diagram of the anodic oxidation of RS-SiC and the removal of oxide is shown in the figure 4. It can be found that all the oxides are completely removed, and the surface morphologies in the figure 3(a) are similar with those in the original RS-SiC surface. Meanwhile, the surface morphologies obtained by SEM under the high magnification in the figure 3(b) indicate that there are some tiny scratches on the polished surface. We hypothesis there are two reasons for generation of these tiny scratches. Firstly, the initial RS-SiC surface has scratches which are generated by the diamond lapping process, and these scratches are filled and closed by the volume expansion of the oxides in the anodic oxidation process. However, during polishing process, these regions have a relatively higher micro-MRR; thus, the scratches are revealed. Nevertheless, sizes of them are reduced, as shown in the figure 4. Secondly, distribution of the oxide on the oxidized RS-SiC surface is asymmetric among different Si grains and different SiC grains, and there are plenty of cracks and projections on the oxidized surface, especially at the oxides generated from initial SiC grains, which are common characters in the anodic oxidation of RS-SiC. So polishing of the oxide layer is unavoidable to generate scratches, because the material removal is realized by the abrading between the oxide and the nano-scale ceria particles. Although tiny scratches are inevitable generated in combination of the anodic oxidation of RS-SiC and the abrasive polishing of the oxide layer, sizes and depths of these scratches are reduced relative to those on the initial RS-SiC surface obtained by diamond lapping in figure 2(a).
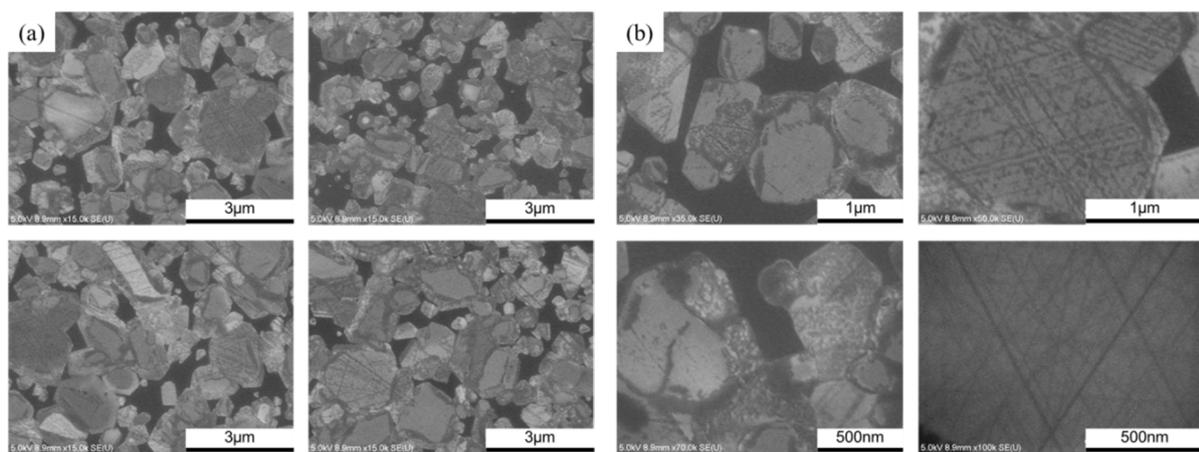

Figure 3. Surface morphologies of the polished RS-SiC samples obtained by the SEM observation. (a) under low magnification. (b) under high magnification.
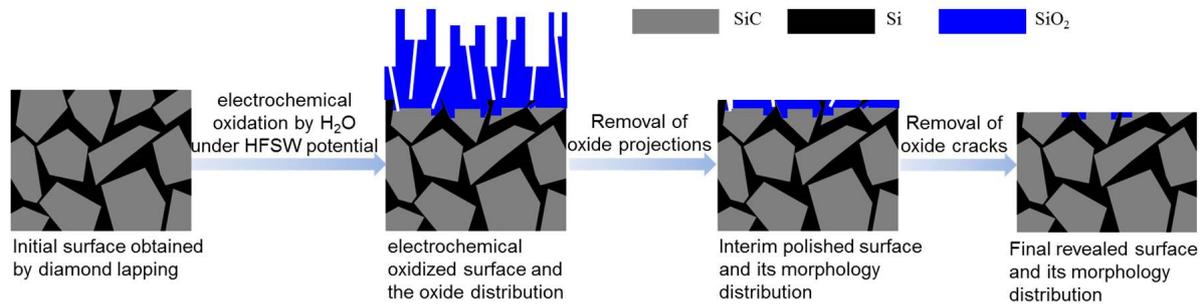
Figure 4. Schematic diagram of anodic oxidation of the RS-SiC and removal process of the oxide.

For the purpose of quantitatively analysis of the polishing result, the polished RS-SiC surface is investigated by the SWLI measurement, and the results are shown in figure 5. After abrasive polishing, the surface roughness rms is 3.688nm, as shown in figure 5(a). Comparing with surface roughness rms 189.004nm of the anodic oxidized RS-SiC surface shown in the figure 2(c), the ceria slurry polishing process greatly improves the surface quality. Although it is no better than that of the initial surface roughness 2.814nm in figure 2(a), there is no visible scratches on the polished surface, which can also be verified by the surface profile in the figure 5(b).
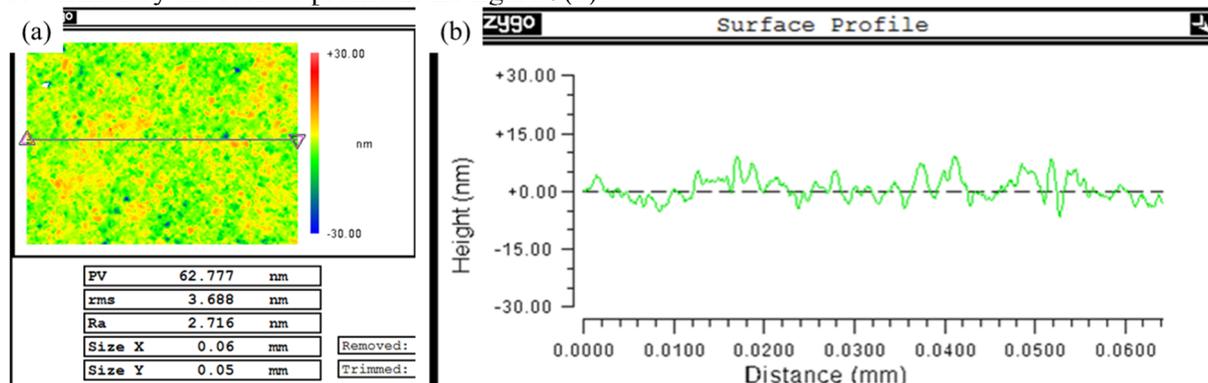

Figure 5. Quantificational analysis of the polishing result by the SWLI measurement. (a) surface morphology and surface roughness. (b) character of the cross-sectional profile.

4. Conclusions

Machinability of the oxide layer obtained in anodic oxidation of RS-SiC by pure-water is investigated by ceria slurry polishing in this study. The following conclusions can be obtained.

(1) Quantitative analysis of the surface quality of the oxidized RS-SiC sample has been conducted by the SWLI measurement. Along with the increase of oxidation time, surface quality of the oxidized RS-SiC is changing better at the beginning, and rapidly deteriorated in the further oxidation process.

(2) Machinability of the oxide layer has been validated by the abrasive polishing of the oxidized RS-SiC. After the abrasive polishing, the obtained surface roughness rms is 3.688nm, which is better than the surface roughness rms 189.004nm of the oxidized RS-SiC sample. What's more, there is no visible scratch on the polished surface, which indicates that this anodic oxidation method is feasible.

Acknowledgments

References

[1]   Shen X M, Deng H, Zhang X N, Peng K, Yamamura K 2016 Preliminary study on atmospheric-pressure plasma-based chemical dry figuring and finishing of reaction-sintered silicon

carbide Optical Engineering 55(10) 105102

[2]   Shimozono N, Shen X M, Deng H, Endo K and Yamamura K 2015 Figuring and Finishing of Reaction-Sintered SiC by Anodic Oxidation Assisted Process Key Engineering Materials 625 570-575

[3]   Peng K, Shen X M, Yamamura K, Zhang X N, Wang D 2017 Investigation on the formation of projections and cracks in anodic oxidation of reaction-sintered silicon carbide IOP Conference Series: Materials Science and Engineering 167(2017) 012064

[4]   Watanabe J, Touge M, Sakamoto T 2013 Ultraviolet-irradiated precision polishing of diamond and its related materials Diamond and Related Materials 39 14-19

[5]   Ishimaru D, Touge M, Muta H, Kubotaa A, Sakamoto T, Sakamoto S 2012 Burr Suppression Using Sharpened Pcd Cutting Edge by Ultraviolet-Ray Irradiation Assisted Polishing Procedia CIRP 1(1) 184-189

[6]   Shen X M, Dai Y F, Deng H, Guan C L, Yamamura K 2013 Ultrasmooth reaction-sintered silicon carbide surface resulting from combination of thermal oxidation and ceria slurry polishing Optics Express 21(12) 14780-14788

[7]   Song Y, Dhar S, Feldman L C, Chung G, Williams J R 2004 Modified Deal Grove model for the thermal oxidation of silicon carbide Journal of Applied Physics 95(9) 4953-4957

[8]   Shen X M, Yamamura K, Zhang X N, Zhang X P, Wang D, Peng K 2016 Research on optimal process parameters in thermally oxidation-assisted polishing of reaction-sintered silicon carbide Proceedings of SPIE 9683 96832B

[9]   Deng H, Yamamura K 2012 Smoothing of reaction sintered silicon carbide using plasma assisted polishing Current Applied Physics 12(3) S24-S28

[10] Shen X M, Tu Q Z, Deng H, Jiang G L, Yamamura K 2015 Mechanism analysis on finishing of reaction-sintered silicon carbide by combination of water vapor plasma oxidation and ceria slurry polishing Optical Engineering 54(5) 055106

[11] Jiang G L, Shen X M, Tang J, Zhang X N, Zhang X P, Yamamura K 2016 Oxidation characteristic and machining performance of reaction sintered Silicon Carbide ceramic in Anodically Oxidation-Assisted Polishing International Journal of Anodic Science 11(2016) 1512-1529

[12] Shen X M, Dai Y F, Deng H, Guan C L, Yamamura K 2013 Comparative analysis of oxidation methods of reaction-sintered silicon carbide for optimization of oxidation-assisted polishing Optics Express 21(22) 26123-26135

[13] Tu Q Z, Shen X M, Zhou J Z, He X H, Yamamura K 2015 Efficient processing of reaction-sintered silicon carbide by anodically oxidation assisted polishing Optical Engineering 54(10) 105113.

[14] Shen X M, Tu Q Z, Deng H, Jiang G L, He X H, Liu B, Yamamura K 2016 Comparative analysis on surface property in anodic oxidation polishing of reaction-sintered silicon carbide and single-crystal 4H silicon carbide Applied Physics A: Material Science & Processing 122(4) 354

[15] Li C H, Bhat I B, Rongjun Wang R J, Seiler J 2004 Electro-Chemical Mechanical Polishing of Silicon Carbide Journal of Electronic Materials 5(33) 481-486

[16] Shen X M, Dai Y F, Peng W Q, Nagano M, Yamamura K 2012 The Improvement of Removal Function in Local Wet Etching by using Eccentric Rotation System Key Engineering Materials 516 504-509

[17] Peng W Q, Li S Y, Guan C L, Shen X M, Dai Y F, Wang Z 2013 Improvement of magnetorheological finishing surface quality by nanoparticle jet polishing Optical Engineering 52(4) 043401

[18] Shen X M, Peng K, Zhang X N, Yin Q, Yamamura K 2016 Research on Simultaneous Electrochemical Oxidation-Assisted Polishing of Reaction-Sintered Silicon Carbide by Ceria Slurry Research and Reviews in Electrochemistry 7(2) 101