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Machining Characteristics of Graphene Oxide-Based Nanosuspensions in Abrasive Machining of Single-Crystal Si and SiC

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ABSTRACT: Single-crystal silicon (Si) and silicon carbide (SiC) are core semiconductor materials in communication, lighting, power generation, and transportation. However, their high hardness and wear resistance combined with low fracture toughness have posed significant challenges for high-efficiency and low-damage machining. Aqueous suspensions containing nanoparticle additives have recently been developed for sustainable manufacturing due to their satisfactory tribological performance and environmentally friendly nature. In this work, nanoadditives, including two-dimensional (2D) graphene oxide (GO) nanosheets and zero-dimensional (0D) diamond nanoparticles, were ultrasonically dispersed in water to formulate different GO-based nanosuspensions for achieving high-efficiency and low-damage abrasive machining. The experimental results indicated that GO nanosuspension was a suitable coolant for grinding Si, generating a ground surface of 32 nm in R_a, owing to its great lubricity and excellent resistance against mechanical abrasion. Diamond-GO hybrid nanosuspension demonstrated a synergistic effect in abrasion, lubrication and oxidation, which was thus appropriate for polishing SiC single crystals, leading to approximate 60% and 30% improvements in removal and roughness respectively, in comparison to a commercially available diamond suspension.

Keywords: GO-based nanosuspension; Brittle material; Abrasive machining; Lubrication; Abrasion; Oxidation

1. Introduction

Wear is a long-standing issue in abrasive machining processes, which causes failure of diamond grits and degeneration of surface quality, in combination with lower machining efficiency [1–4]. Oil-based emulsions or lubricants have been substantially employed in grinding to reduce abrasive wear, prolong wheel lifetime, and improve ground surface quality [5–7]. The oil-based grinding fluids, however, often entail severe environmental issues as they often possess inherent toxicity and are non-biodegradable [8–10]. Therefore, considerable efforts have been directed towards developing water-based lubricants to address this issue [11–13]. For applications in sustainable machining, water-based lubricants provide desirable properties such as low-cost and environment-friendly nature, high burning resistance, and great cooling performance. Nevertheless, Kinoshita et al. [12] demonstrated that water alone cannot offer sufficient lubrication due to weak load-carrying capability and low lubricity, and additives including various chemical compounds are thus needed in order to enhance its lubrication performance in many machining contexts. These chemical additives, however, tend to cause environmental pollution during preparation, usage, and disposal [14–16]. As a result, it is essential to develop high-performance aqueous lubricants that not only enhance lubricity during abrasive machining but are also environmentally friendly.

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Novel aqueous suspensions containing nanoadditives such as nano-Al_2O_3 [17], nano-TiO_2 [18], graphene oxide (GO) nanosheets [19], SiO_2-GO hybrid nanoparticles [20], or Al_2O_3-GO hybrid nanoparticles [21] have recently been developed to replace traditional oil-based lubricants in both metal- and ceramic-machining processes [22–26]. For example, Wu et al. [18] formulated aqueous nanosuspensions containing TiO_2 nanoparticles of 25 nm and found that when rolling at temperatures of 850, 950 and 1050 °C, a 4.0 wt.% TiO_2 nanosuspension sprayed onto roll surfaces was conducted comparably to a 1.0 vol.% oil-in-water emulsion in terms of rolling forces and rolled strip surface quality. This was because TiO_2 nanoparticles could act as abrasives to polish work rolls during hot rolling, thereby, improving surface quality with a reduced rolling force. Li et al. [26] prepared an aqueous GO nanosuspension and was supplied in a conventional flood manner for nanogrinding of $Gd_3Ga_5O_{12}$ (GGG) laser crystals, leading to significantly lower surface damage and subsurface defects than an oil-in-water emulsion owing to its reduced interfacial shear force during grinding. Huang et al. [20] developed a novel hybrid nanosuspension through adding 25 to 30 nm SiO_2 nanoparticles into a GO solvent for use in the grinding of optical glass. Their experimental results indicated that using such a hybrid nanosuspension could achieve an improvement in both removal efficiency and ground surface quality due to its synergistic effect in polishing surface asperities and reducing abrasive wear. The effectiveness of GO-based suspensions has also been demonstrated in the lapping of sapphire single crystals [27]. The in-depth investigation indicated that GO nanosheets decorated with abundant oxygen-containing groups were partially reduced under mechanical abrasion, generating free radicals such as hydroxyl radicals. During abrasive machining, those powerful oxidizing agents can react with chemically inert crystals to generate oxide layers on top of surfaces, thereby favoring mechanical removal with improved surface finishing. Apparently, GO-based nanosuspensions have offered promising potential for use in ceramic shaping processes. The performance and mechanism of GO-based nanosuspensions in both grinding and polishing of hard and brittle semiconductor materials is therefore essential to unveil.

In this work, two types of GO-based nanosuspensions were synthesized through ultrasonically dispersing GO nanosheets into water or diamond nanoparticles into GO aqueous solutions. The tribological and machining performance of both developed GO and diamond-GO nanosuspensions were evaluated in the grinding of Si single crystals and polishing of C- and Si-faced SiC single crystals. The machining mechanisms involved in these processes were unveiled.

2. Experimental Details

2.1. Materials and Mechanical Properties

The workpiece materials used for grinding and polishing were commercially available single-crystal Si, C- and Si-faced SiC substrates, respectively, which were 10 mm in width, 10 mm in length, and 0.3 mm in thickness. The initial surface roughness of those as-received Si and Si-faced SiC specimens was approximately 0.2 nm in S_a, which was measured with an atomic force microscope (AFM, Dimension Icon, Bruker, USA). The as-received C-faced SiC wafers, however, had a slightly rougher surface of 0.5 nm in S_a. The hardness and elastic modulus of these as-received Si, C- and Si-faced SiC single crystals were measured using an in-situ nanoindenter (ALEMNIS, Alemnis AG, Thun, Switzerland). As shown in Figure 1, nanoindentation of an applied load of 60 mN was conducted using a load-function including 10 s loading, 10 s holding, and 15 s unloading [28]. The corresponding material properties are listed in Table 1.

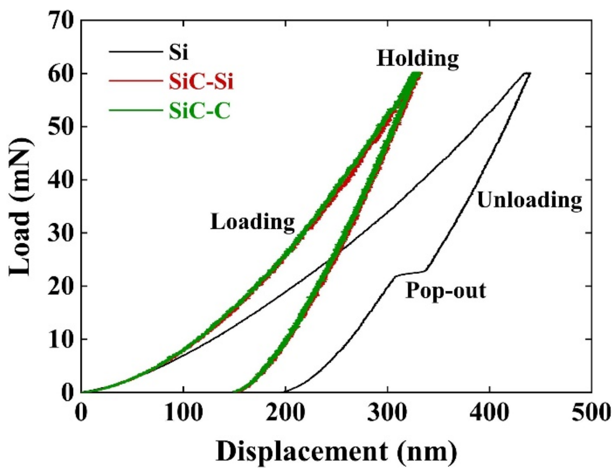

Figure 1. Typical *P-h* curves of nanoindentations conducted on (001)-faced Si, Si- and C-faced SiC single crystals at an indentation load of 60 mN.

Table 1. Properties of as-received Si and SiC single crystals.

Material	Young's Modulus E (GPa)	Hardness H (GPa)	Toughness K_c ($MPa.m^{1/2}$)	Roughness S_a (nm)
(001)-Si	181	12	0.7	0.2
Si-faced SiC	376	35	3.3	0.2
C-faced SiC	420	38	2.7	0.5

In this study, GO nanosheets of multi-layered structures were used to formulate GO nanosuspensions, which had an oxidation degree of around 45%. Single-crystal diamond particles of 500 and 1000 nm in average diameter were employed to prepare both diamond and diamond-GO nanosuspensions. The laminar GO nanosheets have wrinkles on their surface, as displayed in Figure 2a. The diamond particles exhibit irregular morphologies and sharp edges but have relatively uniform size distribution, as shown in Figure 2b,c.

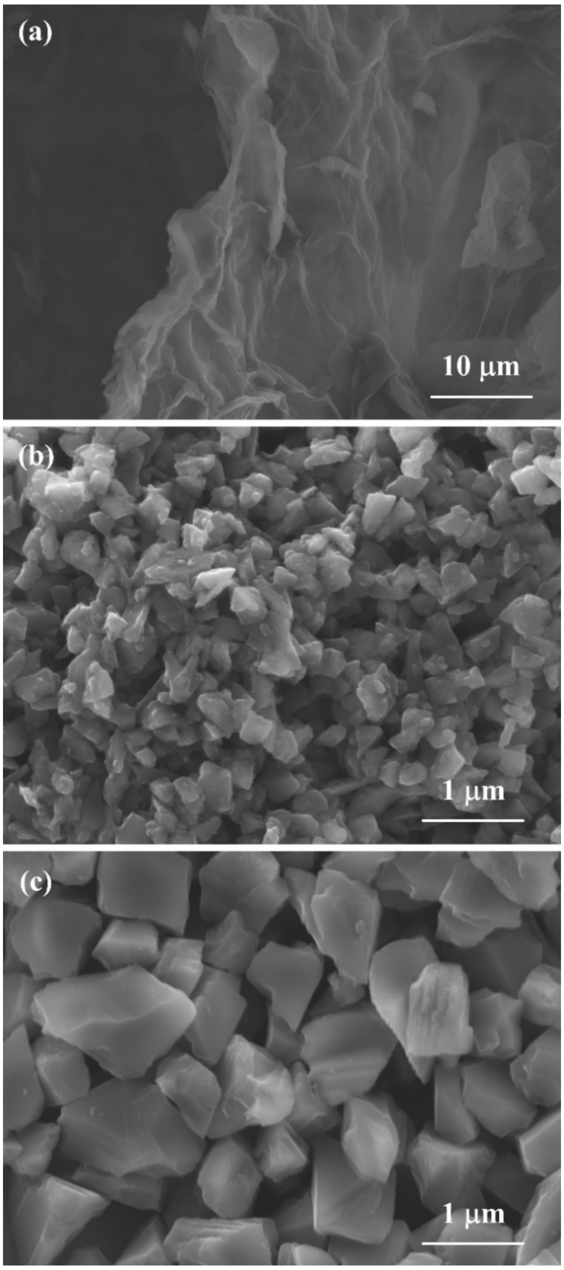

Figure 2. SEM micrographs of (**a**) graphene oxide nanosheets and diamond grits of size of (**b**) 500 nm and (**c**) 1000 nm.

2.2. Formulation of GO and Diamond-GO Nanosuspensions

To formulate aqueous GO nanosuspensions, GO nanosheets were dispersed into deionized water under ultrasonication of 350 W at an additive content of 0.50 wt.%. They were then diluted to obtain GO solutions of 0.025, 0.05, 0.10 and 0.20 wt.%, as GO is capable of offering sufficient lubricity when its content reaches 0.05 wt.% [29]. For comparison, water and commercial emulsion were also employed as benchmarks. To prepare hybrid nanosuspensions of diamond particles and GO nanosheets, diamond nanoparticles were added in a 0.25 wt.% GO solution at a fixed content of 2.0 wt.%, followed by de-agglomeration with a high-power ultrasonic probe of 350 W for 10 min. Diamond nanoparticle aqueous suspensions of sizes of 500 and 1000 nm were also prepared, which had a fixed additive content of 2.0 wt.%. In addition, commercial diamond abrasive suspensions of 500 and 1000 nm were also employed for comparison.

2.3. Grinding with GO Aqueous Nanosuspensions

Grinding tests of an up-grinding mode were conducted on an ultra-precision surface grinder (Okamoto UPZ315Li, Japan). A resin-bonded diamond grinding wheel (SD800, Asahi Diamond, Yamanashi, Japan) with a diameter of 180 mm and a width of 5 mm was used. The SD800 grinding wheel has a mean abrasive grit size of 20 μm. During grinding, distilled water, oil-in-water emulsion, and GO nanosuspensions were individually fed into the grinding area at a 6 L/min flow rate for comparison. Grinding forces were obtained by means of a dynamometer (9275B, Kistler, Winterthur, Switzerland). The friction coefficient of grinding, μ, was calculated by an equation, $\mu = F_t/F_n$, where F_t is tangential force and F_n is the normal force. The depth of cut (DOC) and wheel speed were varied from 40 to 80 μm and 40 to 80 m/s, and table speed was fixed at 500 mm/min, respectively. After testing, ground Si substrates were analyzed using a white light interferometer (SuperView W1, Chotest, Shenzhen, China) and a scanning electron microscope (FE-SEM, Zeiss Sigma 500, Göttingen, Germany). Note that the surface roughness R_a was measured perpendicular to the grinding direction.

2.4. Polishing with Diamond-GO Nanosuspensions

The polishing performance of Si- and C-faced SiC single crystals with diamond-GO hybrid nanosuspensions was evaluated using a precision polishing machine (Smoothneer-6, Truer, Shanghai, China). A polishing plate made of woven silk cloth was used. The polishing load was 5 N, the polishing speed was 150 rpm, the suspension flow rate was 50 mL/h, and the polishing duration was fixed at 60 min. Polishing with diamond-GO nanosuspensions, diamond nanoparticle water suspensions, and commercial diamond abrasive suspensions are named as chemically enhanced polishing (CEP), mechanical polishing (MP), and mechanical-chemical polishing (MCP), respectively. The detailed experimental information is listed in Table 2. After testing, polished SiC substrates were analyzed using an X-ray photo spectroscopy (XPS, Thermo Scientific K-Alpha, Carlsbad, USA). The white light interferometer and FE-SEM were used to measure surface roughness (S_a) and morphologies, respectively. Note that the S_a values were measured over an area of 50 × 50 μm^2.

Table 2. Conventional and chemically enhanced polishing processes for machining of SiC single crystals.

<table>
<tr><th>Polishing Process</th><th>Slurry</th><th>Abrasive</th><th>Grit Size (nm)</th><th>Content (wt.%)</th></tr>
<tr><td>Mechanical polishing (MP)</td><td>Diamond water suspension</td><td rowspan="3">Monocrystalline diamond</td><td rowspan="3">500
1000</td><td>2.0</td></tr>
<tr><td>Mechanical-chemical polishing (MCP)</td><td>Commercial diamond suspension (DS002, Truer)</td><td>2.0</td></tr>
<tr><td>Chemically enhanced polishing (CEP)</td><td>Diamond-GO hybrid nanosuspension</td><td>2.0</td></tr>
</table>

3. Results and Discussion

3.1. Grinding Performance

The friction coefficients of grinding, *i.e.*, force ratios, generated using GO suspension with different additive contents are shown in Figure 3. The results produced with both water and oil-on-water emulsion were also plotted for comparison. The GO suspensions of different tested concentrations yielded lower force ratios than both water and emulsion, as shown in Figure 3, indicating improved interfacial lubrication. The achievement of significantly smaller friction coefficients is because GO nanosheets possess great lubricity as a result of their inherent layered structure, as well as superior resistance against mechanical abrasion, as reported in [26]. It is also seen in Figure 3 that the force ratio decreases with the increase of GO content to 0.1 wt.%, and further increase leads to a slight increase in force ratio. This was because exceeded nanoadditives in water tend to agglomerate, which might cause unnecessary stress concentration and thereby, an increase in force ratio. The 0.1 wt.% GO nanosuspension generated a relatively better performance in lubrication and was hence selected for further grinding tests.

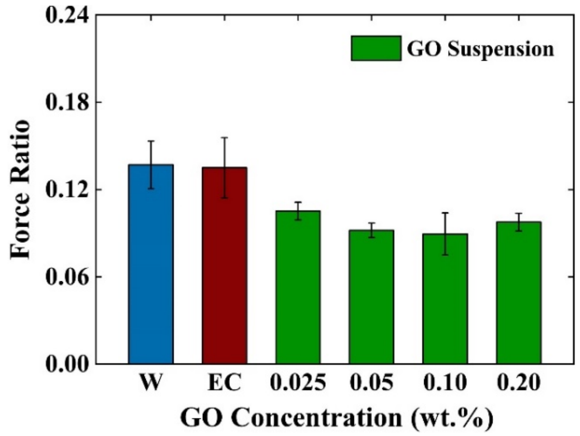

Figure 3. Effect of GO content on force ratio of the Si single crystals ground using GO nanosuspensions at a DOC of 60 mm, wheel speed of 60 m/s, and a table speed of 500 mm/min. The results obtained from water (W, blue bar in Figure 3) and emulsion coolant (EC, red bar in Figure 3) were used for comparison.

The effects of DOC and wheel speed on the specific tangential force of the single-crystal Si ground with water, emulsion, and 0.1 wt.% GO nanosuspension is displayed in Figure 4. For all three tested coolants, a higher DOC value led to larger specific tangential forces. The increase in DOC produced a greater chip thickness, which hence resulted in a higher grinding force [30]. As displayed in Figure 4b, the influence of wheel speed on the specific tangential force is inconsistent with the DOC effect for all the tested coolants. The tangential force decreases when a faster wheel speed is used, owing to the smaller undeformed chip thickness at higher speeds. As expected, using 0.1 wt.% GO nanosuspension generated significantly lower tangential force than both water and emulsion coolants for all the tested DOC and wheel speed values. These results clearly indicate that grinding with 0.1 wt.% GO nanosuspension achieved an improved performance, mostly likely attributed to its great lubricity.

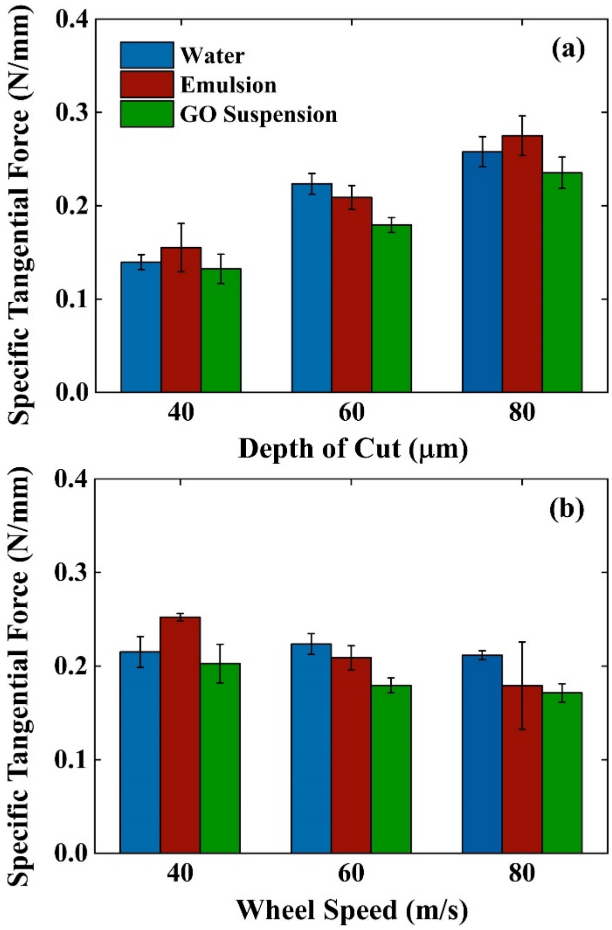

Figure 4. Effects of (**a**) depth of cut (DOC) and (**b**) wheel speed on the grinding force during grinding Si single crystals using pure water, oil-in-water emulsion coolant, and 0.1 wt.% GO nanosuspension at a table speed of 500 mm/min.

The effects of DOC and wheel speed on surface roughness of the Si single crystals ground using water, emulsion, and 0.1 wt.% GO nanosuspension is shown in Figure 5. For all three tested coolants, the increase in DOC from 40 to 80 mm resulted in a rougher ground surface, as a higher DOC would produce a greater depth of ploughing of diamond grits. Figure 5b presents that a faster wheel speed generated smaller surface roughness, which was most likely due to an improved rubbing effect and smaller undeformed chip thickness [31]. It is clearly seen that 0.1 wt.% GO nanosuspension resulted in better ground surfaces than water and conventional emulsion coolants, demonstrating its effectiveness in improving grinding performance.

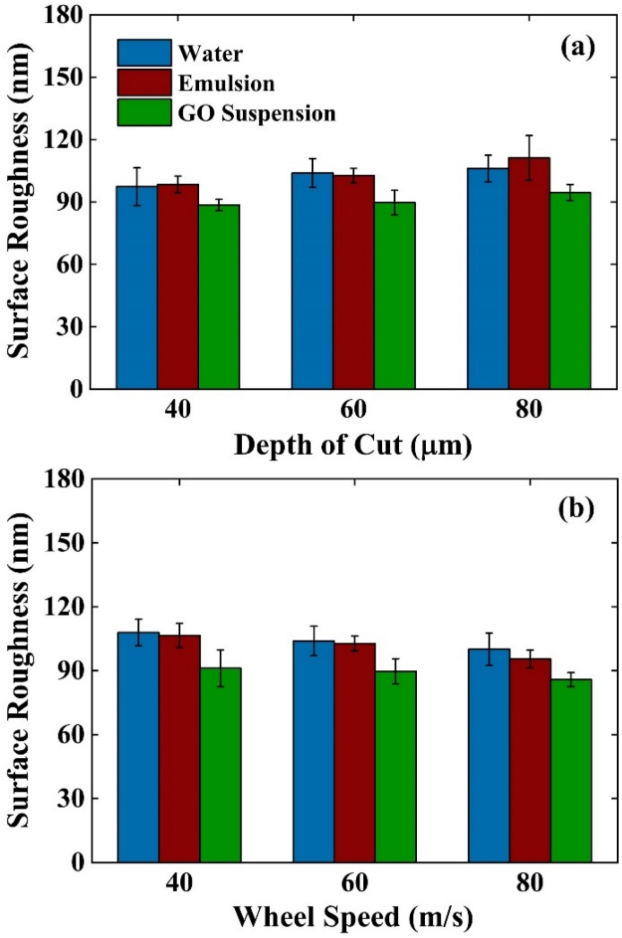

Figure 5. Effects of (**a**) DOC and (**b**) wheel speed on surface roughness (R_a) during grinding Si single crystals using pure water, oil-in-water emulsion coolant, and 0.1 wt.% GO nanosuspension at a table speed of 500 mm/min.

Figure 6 depicts the SEM images of the ground Si surfaces obtained with water and 0.1 wt.% GO suspension. The typical fracture-induced surface damage, including cracks, chips and pits, in combination with ploughing striations, is clearly observed on both ground surfaces. The results clearly indicate that with a 20 μm diamond grit grinding wheel, Si single crystals were removed in a combined manner of brittle and ductile removal [32,33]. The ground surface was achieved with 0.1 wt.% GO suspension, however, presents more ploughing striations and is smoother than that produced with water, as illustrated in Figure 6b, demonstrating improved performance in surface quality.

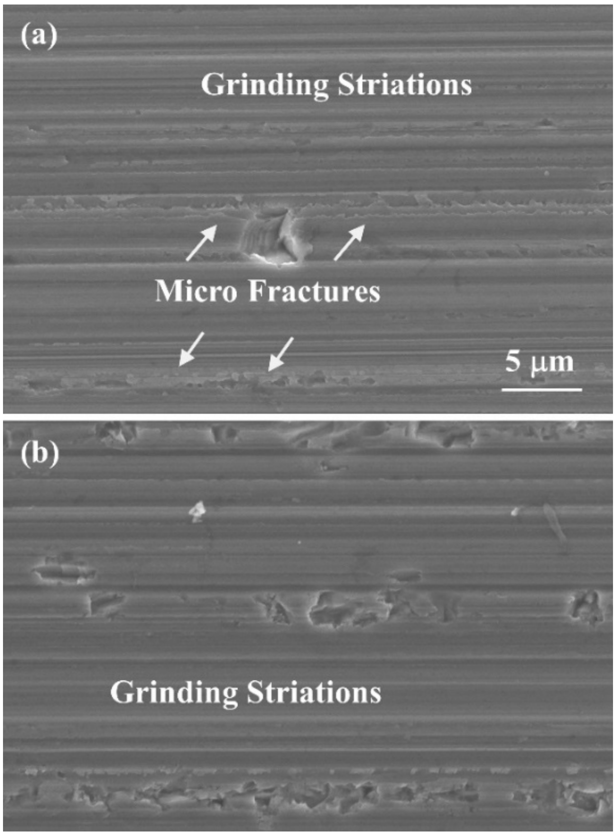

Figure 6. SEM images of the single-crystal Si surface ground using (**a**) water and (**b**) 0.1 wt.% GO suspension; both images are displayed in the same magnification.

3.2. Polishing Performance

Figure 7 presents the material removal rate of the Si- and C-faced SiC single crystals polished with diamond water (MP), commercial diamond suspension (MCP), and diamond-GO nanosuspension (CEP) at diamond particle sizes of 1000 nm (rough polishing) and 500 nm (fine polishing). The decrease in grit size resulted in a reduction in polishing efficiency for all three types of suspensions. Still, all the removal rate values were greater than those produced using chemical-mechanical polishing (CMP) for both Si- and C-SiC substrates. It is seen from Figure 7 that the three suspensions all produce a relatively higher removal rate when polishing Si-faced SiC, which is most likely due to the lower hardness of the (0001) lattice plane [34]. As expected, CEP improves removal efficiency significantly, as shown in Figure 7a, leading to a 57.7% higher removal rate than MCP but a relatively lower removal rate than MP. When polishing C-SiC, however, CEP performs comparably to MP regarding removal efficiency, with an increase of 75.6% compared to MCP, as depicted in Figure 7b.

Figure 8 depicts the surface roughness of the Si- and C-faced SiC single crystals polished with MP, MCP, and CEP at diamond particle sizes of 1000 nm (rough polishing) and 500 nm (fine polishing). As displayed in Figure 8a,b, C-faced SiC single crystal yielded better polishing quality, and a smaller grit size resulted in a smoother polished surface for the three tested suspensions. All the roughness values of CEP were greatly smaller than those produced using MP and MCP for both Si- and C-faced SiC single crystals. In particular, CEP conducted comparably to CMP regarding surface roughness when polishing C-SiC in the fine polishing context. The experimental results shown in Figure 8 clearly indicate that polishing with hybrid nanosuspensions achieved an improved performance, leading to an increase of around 50% in polishing efficiency and an enhancement of 25% in surface quality compared to those commercially available diamond abrasive suspensions.

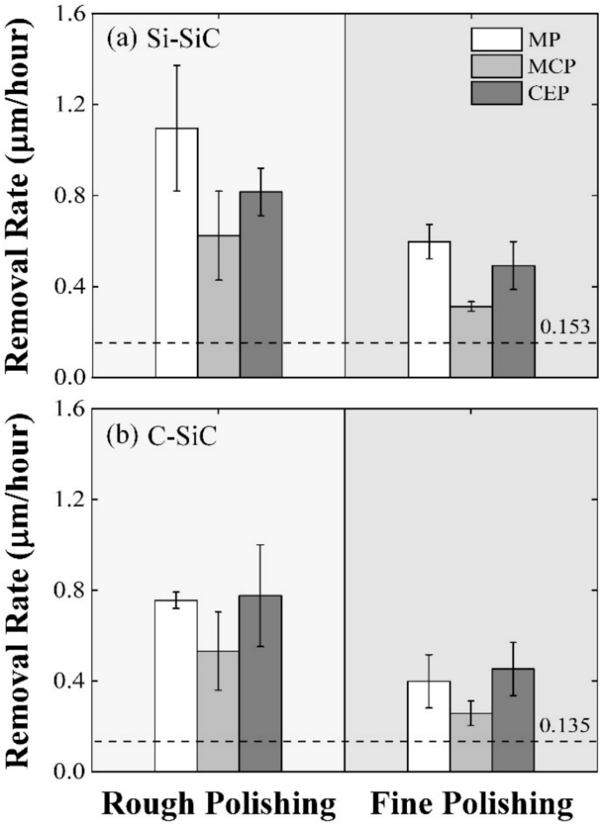

Figure 7. Material removal rate of the (**a**) Si-faced and (**b**) C-faced silicon carbide substrates produced using MP, MCP and CEP processes under rough and fine polishing contexts. The removal rates of Si-faced silicon carbide and C-faced silicon carbide (horizontal lines in **a** and **b**) generated using the CMP process were used for comparison.

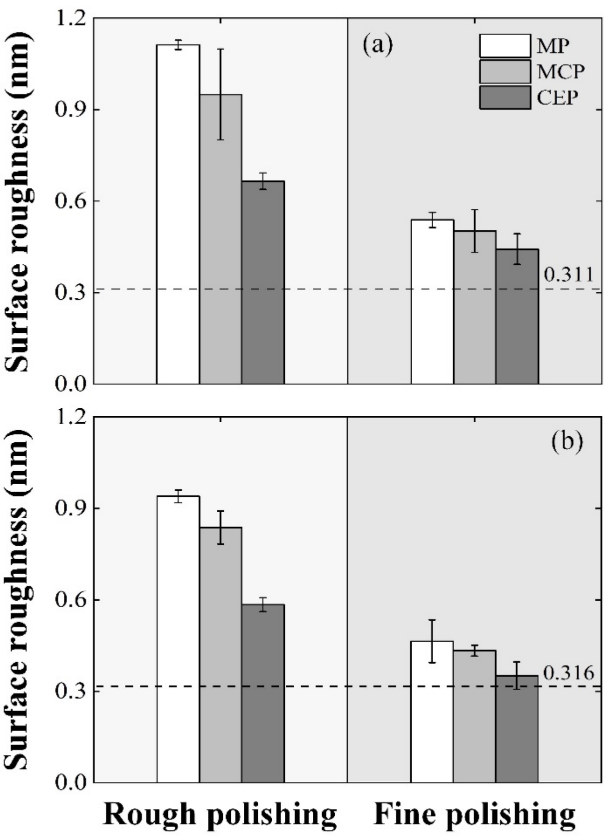

Figure 8. Surface roughness (S_a) of the (**a**) Si-faced and (**b**) C-faced silicon carbide substrates produced using MP, MCP and CEP processes under rough and fine polishing contexts. The roughness values (horizontal lines in **a** and **b**) were obtained using CMP process.

The SEM images of the representative Si-faced SiC surfaces polished with MP, MCP and CEP processes at 500 and 1000 nm diamond grit sizes are shown in Figure 9. No defects of brittle removal, such as cracks, chips and fractures, were found on the three polished surfaces. However, ploughing striations of ductile removal are clearly seen and appear smaller when 1000 nm diamond grits were used, as shown in Figure 9. The surfaces produced by CEP present much fewer striations than those achieved using MP and MCP, and particularly, few striations are observed when 500 diamond grits were used, as shown in Figure 9d, indicating the improved polishing performance with the diamond-GO hybrid nanosuspension.

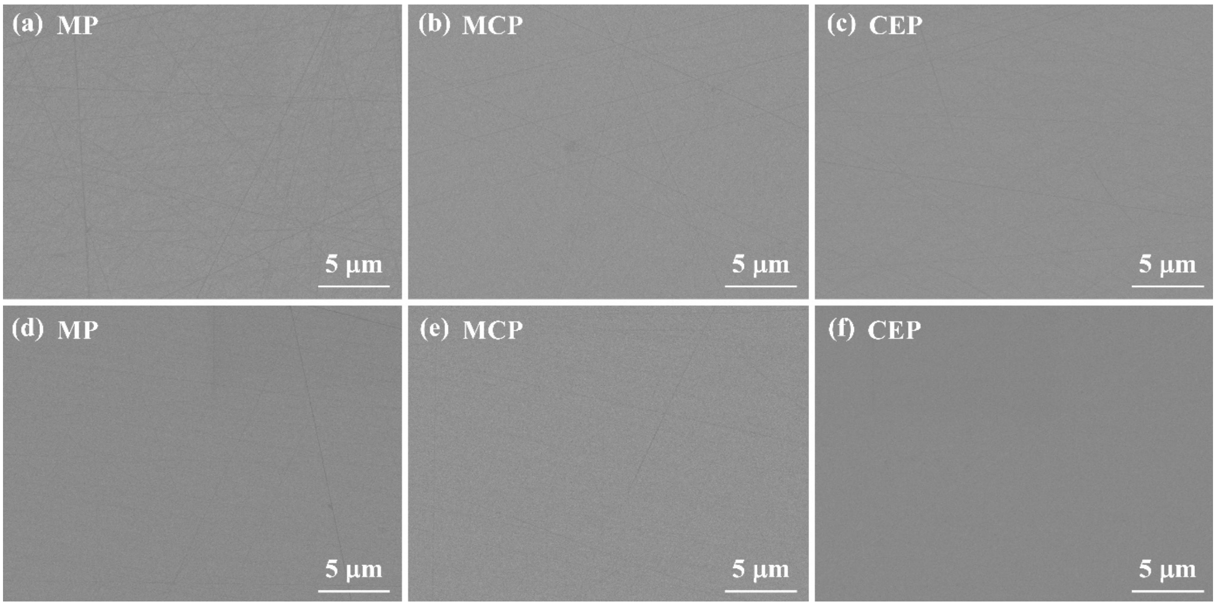

Figure 9. SEM images of the Si-faced SiC single crystals polished using MP, MCP and CEP processes under (**a**–**c**) rough and (**d**–**f**) fine polishing contexts.

3.3. Machining Mechanism of GO-Related Nanosuspensions

Figure 10 presents the Si 2p spectra of the Si-faced SiC surface processed by MP and CEP. In Figure 1a, two strong peaks of Si $2p_{3/2}$ and Si $2p_{1/2}$ of SiC at 100.3 eV and 100.9 eV are clearly observed, respectively. Two weak Si peaks of $SiOC_3$ and SiO_2C_2 are also found at 101.6 eV and 102.6 eV. The CEP-processed SiC surface also detected these two typical Si $2p_{3/2}$ and Si $2p_{1/2}$ peaks of SiC at 100.3 and 100.9 eV, as shown in Figure 10b. In addition, Si peaks of $SiOC_3$ and SiO_2C_2 at 101.4 and 102.2 eV were respectively observed. Meanwhile, in Figure 10b, an extra peak of Si in SiO_3C is clearly found at 102.9 eV. The XPS analysis shown in Figure 10 clearly demonstrates that a chemically reacted layer was formed on Si-faced SiC topmost surfaces during both diamond mechanical polishing and GO-added chemically enhanced polishing processes. The layer has a thickness value varied from 2 to 5 nm, as reported in [34]. Therefore, AFM-based indentation was conducted to explore the deformation characteristics of the chemically reacted layers generated from the polishing processes. As depicted in Figure 11, the chemical reactants produced using the diamond-GO hybrid nanosuspension have a higher indentation depth than that generated using the diamond water suspension, indicative of severe deformation (*i.e.*, lower hardness). The softer layer favours mechanical removal and hence results in improved polishing performance.

Overall, due to its layered structure and oxidation properties as a result of the formation of free radicals under mechanical abrasion, the addition of GO nanosheets could simultaneously enhance the lubrication and oxidation performance of diamond nanoparticle suspensions. This thereby not only relieved the shear force involved in the polishing of single-crystal SiC, preventing the severe scratches caused by relatively large diamond particles but also promoted the extent of the chemical reaction during the polishing, resulting in improved removal efficiency.

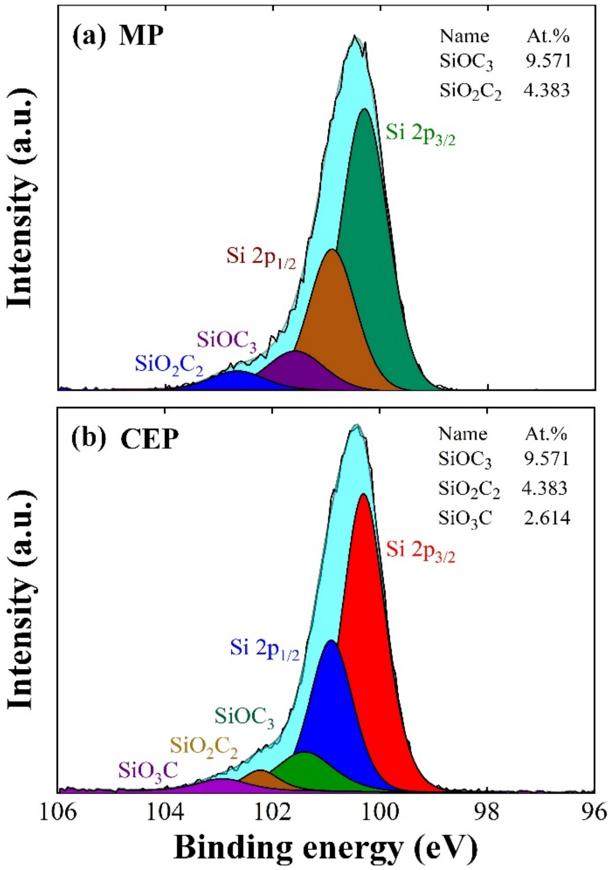

Figure 10. High-resolution XPS Si 2p spectra of the polished Si-faced SiC single crystals using (**a**) MP and (**b**) CEP processes at a fine polishing condition.

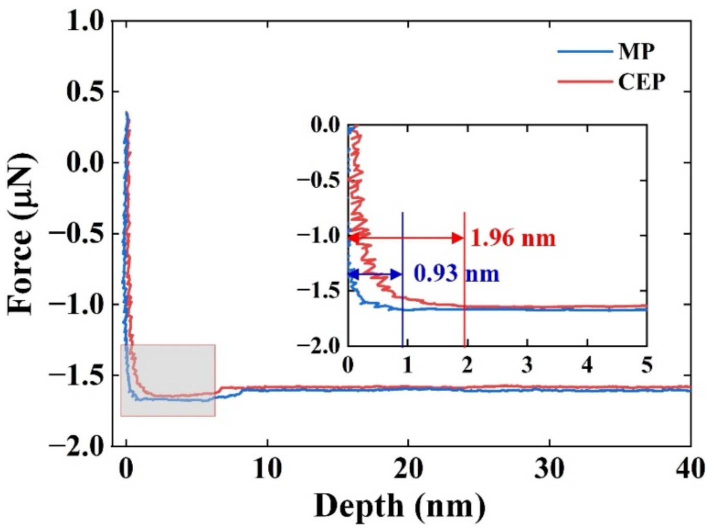

Figure 11. AFM F-D curves of the polished Si-faced SiC single crystals using MP and CEP processes at a fine polishing condition.

4. Conclusions

Aqueous nanosuspensions with GO nanosheets or diamond-GO hybrid nanoparticles as additives were synthesized in this study. The GO nanosuspensions demonstrated a superior lubrication performance to water and a conventionally used oil-in-water emulsion during grinding Si single crystals. The improved grinding performance was likely attributed to the formation of a GO lubricating film between the grinding wheel and work material interface, which provided low resistance for diamond abrasives to shear in the grinding zone. The diamond-GO hybrid nanosuspensions demonstrated a superior synergistic performance during polishing SiC single crystals. The improved polishing performance was due to the synergistic action of the oxidation and lubrication produced by GO nanosheets, in combination with the mechanical removal of diamond nanoparticles.

Author Contributions

Conceptualization, G.Z., S.L. and X.L.; Methodology, G.Z., S.L. and X.L.; Software, L.X. and B.G.; Validation, S.H., L.X. and B.G.; Formal Analysis, G.Z., S.L. and X.L.; Investigation, G.Z., S.L. and X.L.; Resources, S.H. and C.H.; Data Curation, D.Z.; Writing—Original Draft Preparation, G.Z. and S.L.; Writing—Review & Editing, S.H. and Y.W.; Visualization, G.Z., S.L. and X.L.; Supervision, S.H.; Project Administration, S.H.; Funding Acquisition, S.H. and L.X.

Ethics Statement

Not applicable.

Informed Consent Statement

Not applicable.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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