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Investigation of Polishing Pads Impregnated with Fe and Al_2O_3 Particles for Single-Crystal Silicon Carbide Wafers

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Abstract: This study focuses on the development of a novel polishing pad for SiC wafers. Fe and Al_2O_3 particles were impregnated in a polyurethane matrix, thus forming a fixed abrasive polishing pad. Four types of pads with different compositions of Fe and Al_2O_3 were fabricated. A combination of loose and fixed polishing methods was used for polishing with the fabricated pads and was investigated to improve the polishing process. The surface characteristics of the polished SiC wafer and the SiC removal rate during polishing using the designed pads were examined and compared with those for SiC polished with a conventional polyurethane pad. Experimental results showed that the removal rate for SiC in the case of polishing with the pads consisting 1 wt % Fe and 3 wt % Al_2O_3 particles was approximately 73% higher than that observed when polishing using the conventional polyurethane polishing pad. Additionally, the surface roughness of the resulting SiC wafers after polishing with the Fe and Al_2O_3-impregnated pads was identical to that when using the conventional polyurethane pad, without any surface damage. The results indicated that the Fe and Al_2O_3-impregnated pads can be effectively used for SiC wafer polishing. When the proposed process was employed for polishing single-crystal SiC, both the polishing time and cost were reduced. This novel design can facilitate the extensive use of single-crystal SiC wafers in the future.

Keywords: chemical mechanical polishing; single-crystal silicon carbide; polishing pad

1. Introduction

Single-crystal silicon carbide (SiC) is widely used due to its excellent electrical, thermal, and mechanical properties resulting from a variety of features, which include a wide energy band gap, excellent thermal conductivity, high saturated electron drift velocity, and satisfactory chemical stability [1]. SiC is particularly suitable for high-performance devices such as high-power, high-temperature, and high-frequency electronic devices. However, a well-ordered SiC surface and a high removal rate are difficult to achieve due to the mechanical hardness and chemical inertness of SiC [2]. Chemical mechanical polishing (CMP) is the core technology used in semiconductor manufacturing, especially in ultra large scale integration (ULSI) manufacturing. CMP is regarded as the most effective technology to achieve ultra-smoothness without causing surface damage, during the ultra-precise machining of SiC crystal substrates [3,4]. However, the material removal rate (MRR) in CMP is very low. Therefore, new methods must be developed in order to increase the MRR of SiC during polishing process.

Several studies on the CMP of SiC have pursued in order to increase the MRR and verify the polishing mechanisms. Neslen *et al.* [5] studied the effect of process parameter variations on the MRR of SiC by using commercial colloidal silica slurry and found the maximum MRR to be

0.25 mm/h. In another work [6], the chemical and tribological conditions for tribochemical polishing of polycrystalline SiC were investigated in different oxidant solutions, and the surface properties were determined. The results showed that the material removal rate was 200–400 nm/h. Kuo and Currier [7] augmented the MRR in CMP of SiC with an oxidizer-enriched colloidal silica slurry and a hybrid of specially treated sub-micron diamond and colloidal silica slurry (designated as CMP-D slurry). Lee *et al.* [8] used a mixture of a colloidal silica slurry and nanodiamond abrasive (designated as a mixed abrasive slurry (MAS)) to achieve superior MRR and a fine surface. Recent studies have reported that by using mixed abrasives in hybrid CMP, a superior MRR and high scratch reduction could be achieved. The MRR of SiC after CMP with MAS was approximately 550 nm/h. Yamamura *et al.* [9] proposed a novel abrasive polishing technique combined with the irradiation of atmospheric-pressure plasma, called plasma assisted polishing (PAP). This method afforded high-efficiency and high-integrity finishing on difficult-to-machine materials and resolved the low MRR of single-crystal SiC (<0.5 μm/h). Some studies have examined the influence of polishing slurry characteristics, including pH, abrasive size and concentration, rotational velocity of the polishing platen and carrier, and polishing pressure on the MRR of the SiC crystal substrate based on the alumina abrasive in CMP. Kubota *et al.* [10] proposed a surface planarization method for SiC substrates by using Fe abrasive particles and a hydrogen peroxide (H_2O_2) solution. They found that the MRR of the 4H-SiC C-face surface was approximately 800 nm/h at a pressure of 0.06 MPa. Lagudu *et al.* [11] discussed the development of slurries based on silica abrasives that resulted in high amorphous SiC (a-SiC) removal rates (RRs). The ionic strength of the silica dispersion was found to play a significant role in enhancing material removal rate, while also providing very good post-polish surface-smoothness.

This study proposes a novel fixed abrasive polishing pad composed of Fe and Al_2O_3 particles impregnated in a polyurethane matrix. Four types of pads of various compositions, containing 1 wt % Fe particles, 1 wt % Al_2O_3 particles, 1 wt % Fe/1 wt % Al_2O_3 particles, and 1 wt % Fe/3 wt % Al_2O_3 particles, were fabricated. A combination of loose and fixed polishing methods was explored to improve the polishing process. The surface characteristics, surface damage, and removal rate of SiC when using these polishing pads were examined and compared with those observed when using a conventional polyurethane polishing pad. The proposed process is well suited for polishing single-crystal SiC, and it is found to reduce both polishing time and cost.

2. Experimental Section

The nanoparticle-impregnated pads used in this study were made of polyurethane-based (polyester urethane) material, with 1 wt % Fe particles (Chemirite, Ltd., Tokyo, Japan), 1 wt % Al_2O_3 particles (WU-004, Keelung, Taiwan), 1 wt % Fe/1 wt % Al_2O_3 particles and 1 wt % Fe particles/3 wt % Al_2O_3 particles, as shown in Figure 1. The size of the Fe particles is about 500 nm. The size of the Al_2O_3 particles is about 50 nm. The shapes of the Fe and Al_2O_3 particles are nearly circular. The fabrication of the nanoparticle-impregnated pad involved four stages. First, a pad pattern groove was designed using computer aided design (CAD) software (AutoCAD 2015, Autodesk, San Rafael, CA, USA, 2014). Two types of pad patterns were designed in this study, which include concentric and hexagon grooves. Then, a mold was fabricated by computer numerical control (CNC) machining (MV-184, Quaser Machine Tools, Inc., Taichung, Taiwan). In the second step, a mixture of hydrogenated nanodiamond particles was obtained by heat-treating graphite particles under hydrogen atmosphere, which initiates their uniform dispersion into the polyurethane matrix. In the third step, a prototype for the top pad was fabricated. Polyester urethane and nanodiamond were mixed and poured into a mold, which was then compressed to form a cylindrical pad. Finally, the mold and pad were separated to obtain a 5-mm-thick pad prototype with a diameter of 304.8 mm. In the final step, a soft sub-pad was fixed to the prototype of the pad to create a nanoparticle-impregnated polishing pad. A pad consisted of a soft subpad secured to a more rigid top pad to ensure conformability to the wafer surface on a global scale. In this study, four polishing pads and two slurries were used, as shown in Table 1.

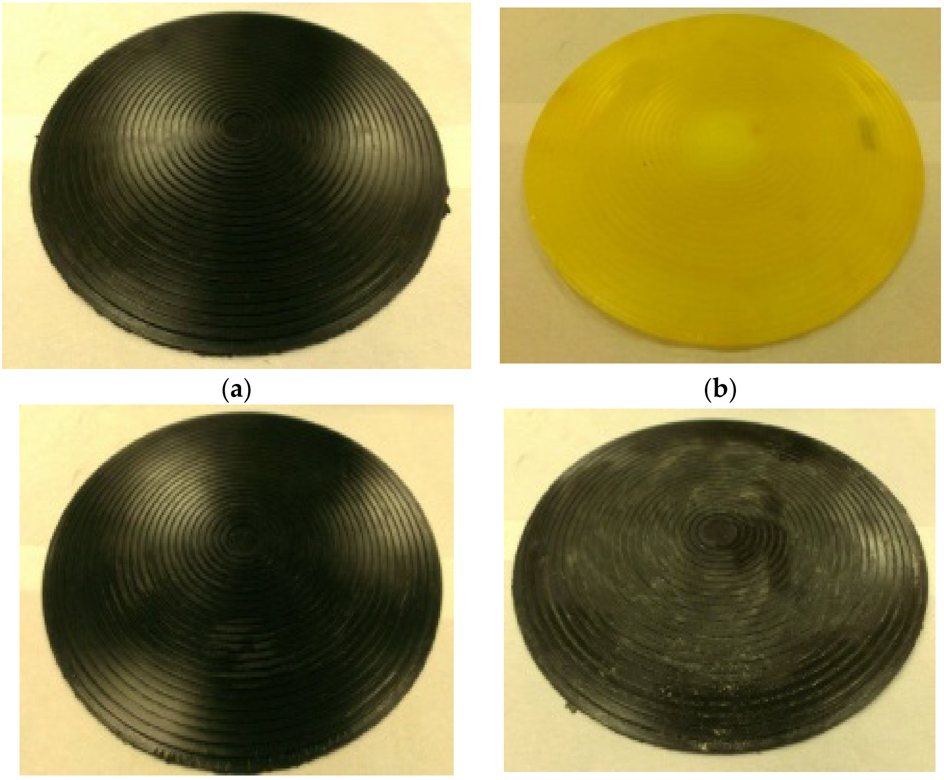
(**a**) (**b**) (**c**) (**d**)

Figure 1. Images of polishing pads impregnated with Fe and Al_2O_3 particles at various concentrations: (**a**) 1 wt % Fe particles; (**b**) 1 wt % Al_2O_3 particles; (**c**) 1 wt % Fe and 1 wt % Al_2O_3 particles; and (**d**) 1 wt % Fe particles and 3 wt % Al_2O_3 particles.

Table 1. Nanoparticle impregnated pads.

Polish Pad	Content	Slurry
Pad-A	1% Fe	H_2O_2 (20%) + HCL (pH ~ 4.0)
Pad-B	1% Al_2O_3	
Pad-C	1% Fe + 1% Al_2O_3	
Pad-D	1% Fe + 3% Al_2O_3	

The polishing and dressing experiments were conducted at various speeds by mounting the polishing pad on a commercially modified, variable-speed dressing machine (Home-Made), as shown in Figure 2. Therefore, the polishing speed was adjustable. Then, the pad was mounted on the polishing plate. During polishing, a power head applied a specific load to SiC having dimensions of Φ100 mm × 0.5 mm. A dispenser was used to supply the slurry. Eight commercially available 50.8 mm, *n*-type, 4H-SiC (0001) off-axis wafers (2" 4H *N*-type 4° off-axis, 440 µm thickness, As-cut SiC wafer-Dummy Grade, MAST Ltd, Hsinchu, Taiwan) were used in this study. The resistivities of these wafers were in the range of 0.015–0.03 Ω·cm, and their micropipe density was less than 15 cm^{-2}. Each wafer was attached to a holder that was connected to the wafer carrier head rotating at 80 rpm. A polyurethane pad was mounted on the table disk with a diameter of 304.8 mm that rotated at 100 rpm. The applied pressure was approximately 3 kg· wt. In the experiments, H_2O_2 (20%) (Chang Chun Group, Ltd, Miaoli, Taiwan) and HCl (pH ~ 4.0) (FU YUAN CHEMICALS CO., LTD., Zhengzhou, China) slurry was used for the novel polishing pad, and $KMnO_4$-based Al_2O_3 slurry was used for the traditional polishing pad (IC1000, Cabot, Boston, MA, USA); the latter is referred to as Type E. The flow rate of the slurry was maintained at 150 mL/h. During conditioning, the diamond pad conditioner was attached to the rotating head (spinning at 100 rpm) via a connecting holder. The oscillation speed of the conditioner was fixed at 5 mm/s, and the applied load was approximately ~3 kg.

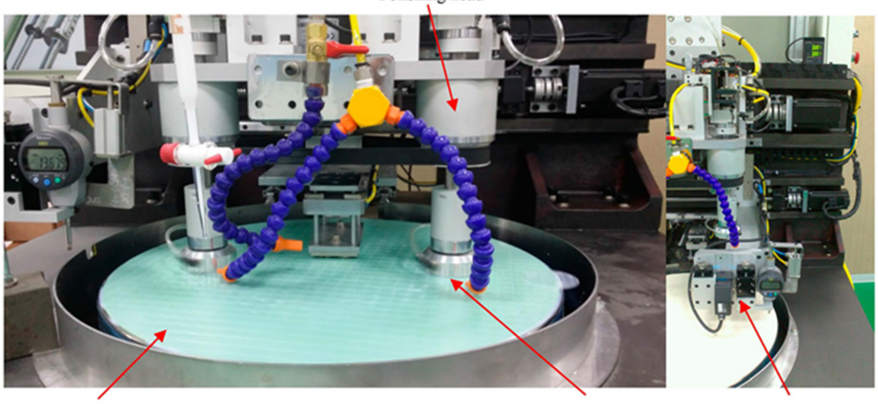

Figure 2. Experimental setup for the polishing system used in this work.

The SiC was polished and the wafer dimensions were determined using non-contact three-dimensional (3D) white light interferometry (New View 8300, Zygo, Middlefield, CT, USA) with an RMS repeatability of 0.01 nm and measuring range of up to 20 mm. Each image was captured over an area of 42 μm × 42 μm of the SiC wafer. The roughness values used in the next section represent the averages of three measured values. The cut rate of the pad was measured using a linear variable differential transformer (LVDT) (543-471B, Mitutoyo, Tokyo, Janpan). The thickness of SiC was measured before and after polishing using a commonly applied system. The measurements were performed at nine positions, with 0.1 μm precision.

3. Results and Discussions

Figure 3 shows variations in the average removal rates of SiC when using the conventional polishing pad (IC1000) and pads impregnated with Fe and Al_2O_3 under identical experimental parameters. Each experiment was repeated thrice to ensure reproducibility of the results. As shown in the figure, the SiC removal rate is higher for the 1 wt % Fe and 3 wt % Al_2O_3-impregnated pads (Pad D) than that for the conventional polyurethane pad. The MRR for the 1 wt % Fe impregnated pad was ~0.33 μm/h, which is higher than that for the 1 wt % Al_2O_3 impregnated pad (~0.18 μm/h). The highest removal rate of ~0.74 μm/h was obtained by using the impregnated pad containing 1 wt % Fe and 3 wt % Al_2O_3. SiC polishing typically involves the formation of an oxide layer due to a chemical reaction, and this layer is removed by the abrasive particles present on the surface of the pad. Thus, the materials on the SiC surface are removed by the combination of chemical reactions and mechanical processes. According to these observations, the Fe-impregnated pad can increase the MRR due to the Fe catalytic reaction in H_2O_2 on the surface of the Fe catalyst. Initially, Fe is immersed in H_2O_2 solution and ionizes to form ferrous ions (Fe^{2+}). Then, Fe^{2+} reacts with H_2O_2 to generate OH radicals ($OH^{\cdot}$), a strong oxidation species. These OH radicals react with the top-surface of the SiC substrate to form an oxide layer when the Fe catalyst comes into contact with SiC. This oxide layer (SiO_2) is mechanically and chemically removed [10]. The Fe and Al_2O_3-impregnated pads tend to increase the propensity for two-body contact interactions, as shown in Figure 4. When the two-body contact dominates the polishing process, the polishing rate of the wafer will be high. For the two-body contact interactions to occur, the abrasive layer must be firmly retained by the pad. The MRRs from the three-body abrasion are lower than those obtained from two-body abrasion because of the presence of loose abrasive particles between the abraded solid surfaces for only ~10% of the time, while they spend ~90% of the time in rolling.

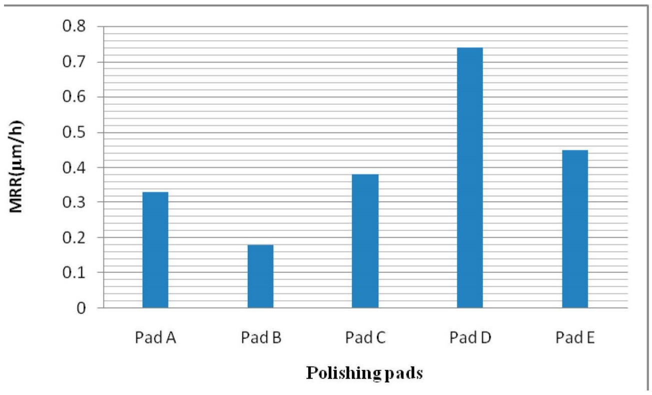

Figure 3. Comparison of material removal rates using different polishing pads.

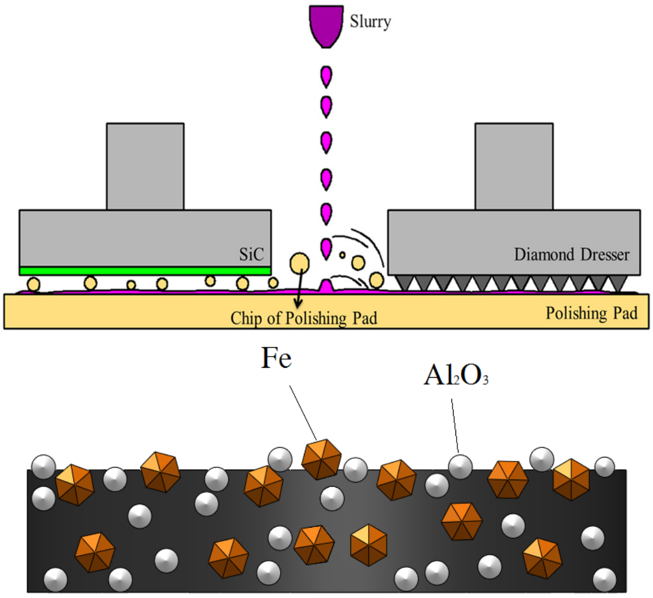

Figure 4. Schematic illustration for the Fe and Al_2O_3-impregnated polyurethane polishing pads.

Figures 5 and 6 show the surface roughness (Ra) of SiC wafers polished by pads impregnated with 1 wt % Fe particles, 1 wt % Al_2O_3 particles, 1 wt % Fe and 1 wt % Al_2O_3 particles, and 1 wt % Fe and 3 wt % Al_2O_3 particles. The surface roughness was measured by Zygo with a measuring window of 42 μm × 42 μm. The results showed that the average Ra values of the SiC samples after polishing were 5.59 Å, 4.66 Å, 2.61 Å and 1.59 Å. Moreover, the surface roughness of the samples polished using the impregnated pads consisting of 1 wt % Fe and 3wt % Al_2O_3 particles was better than that of the samples polished using the other pads. Following Pad E polishing, the average Ra value was determined to be ~1.21 Å. Note that the Ra obtained using Pad D polishing is almost identical to that for Pad E polishing, shown in Figure 6. The MRR for the nanoparticle-impregnated pads is significantly better than that for the polyurethane polishing pad. Hence, pads impregnated with 1 wt % Fe and 3 wt % Al_2O_3 particles can result in a higher MRR while yielding a defect-free surface. After numerous experiments, Bifano *et al.* [12] deduced the critical depth expression for brittle-ductile transition, as given in Equation (1).

$$d_c = \varphi \frac{E}{H} \left(\frac{K_c}{H}\right)^2 \tag{1}$$

where d_c is the critical depth, ψ is a dimensionless quantity, K_c is the fracture toughness, E is the material's Young modulus of elasticity, and H is the hardness. While the relative parameters are configured as K_c = 3 MPa·m$^{1/2}$, E = 430 GPa, and H = 33 GPa, the value of ψ was determined to be 0.15. Following Equation (1), the d_c value for SiC was calculated to be ~16.2 nm. According to the Bifano theory, when the cutting depth (d_e) is lower than d_c, the SiC material removal model suggests the occurrence of ductile behavior; that is, plastic deformation can be expected in this case. Therefore, the value of d_e for diamond penetrating SiC can be calculated using the Equation (2).

$$d_e = \frac{dH_p}{4H} \tag{2}$$

where d_e is the diamond-grit cutting depth, H_p is the pad hardness, H is the SiC hardness, and d is the diameter of the Al_2O_3 abrasive. For this experiment, we used a d value of ~50 nm. Thus, from Equation (2), the d_e value for SiC was calculated to be ~2.4 nm. Therefore, the obtained d_e value is significantly lower than the d_c value, which implies that there are no scratches on the SiC surface after CMP.

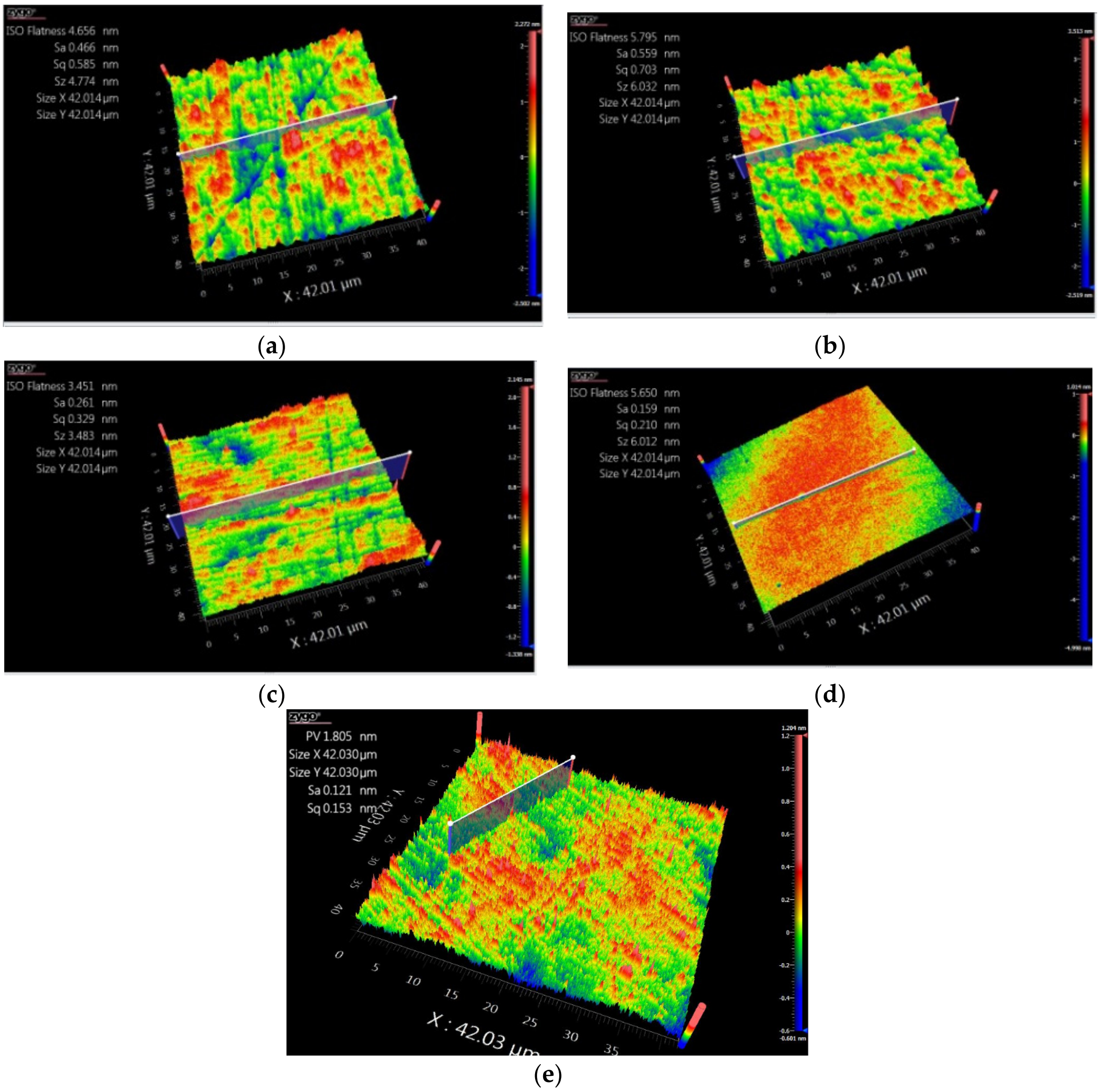

Figure 5. Zygo images of the SiC surface formed after polishing using pads impregnated with (**a**) 1 wt % Fe particles, (**b**) 1 wt % Al_2O_3 particles, (**c**) 1 wt % Fe and 1 wt % Al_2O_3 particles, and (**d**) 1 wt % Fe particles and 3 wt % Al_2O_3 particles. (**e**) Image of the SiC surface when using the traditional polishing pad.

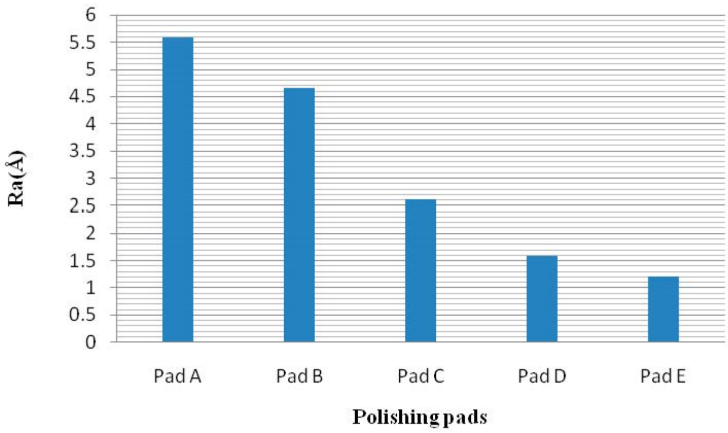

Figure 6. Comparison of the surface roughness (Ra) of SiC wafer after polishing with different polishing pads.

4. Conclusions

In this work, a novel abrasive polishing pad composed of Fe and Al_2O_3 particles impregnated in a polyurethane matrix was developed. Four types of pads, containing 1 wt % Fe particles, 1 wt % Al_2O_3 particles, 1 wt % Fe and 1 wt % Al_2O_3 particles, and 1 wt % Fe and 3 wt % Al_2O_3 particles, were fabricated. A combination of loose and fixed polishing was explored to improve the polishing efficiency. The surface characteristics, surface damage, and MRR of the SiC wafers polished with the fabricated pads were examined and compared with the corresponding attributes observed when using a conventional polyurethane polishing pad. Experimental results showed that the removal rate of SiC with 1 wt % Fe and 3 wt % Al_2O_3 particles was ~73% higher than that obtained by using the conventional polyurethane polishing pad. Further, the surface roughness was almost identical in the samples polished using the Fe and Al_2O_3-impregnated polishing pads and the traditional polyurethane polishing pad, without any surface damage. This indicates that the Fe and Al_2O_3-impregnated pads can be effectively used for SiC wafer polishing. When the proposed process was applied to polish single-crystal SiC, both the polishing time and the cost were reduced. Therefore, this novel pad design and the proposed process reduce the time and cost of polishing, and thus can be used extensively with single-crystal SiC wafers in the future. Also we will plan to carry out further research into the subsurface damage of SiC and the dislocations densities or the surface leakage.

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Conflicts of Interest: The authors declare no conflict of interest.

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