On the Chemo-Mechanical Polishing for Nano-Scale Surface Finish of Brittle Wafers

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Abstract: Chemo-mechanical polishing (CMP) has been a common method to produce nano-scale surface finish of brittle wafers. This paper provides a relatively comprehensive review on the CMP of silicon, silicon carbide and sapphire including both patents and papers. The discussion includes the limitations and further research directions of the CMP technology, the material removal mechanisms, and the control and optimization of the CMP for brittle wafers. The paper concluded that the usage of mix- or coated- abrasives may improve the CMP in terms of less subsurface damage and higher material removal rate.
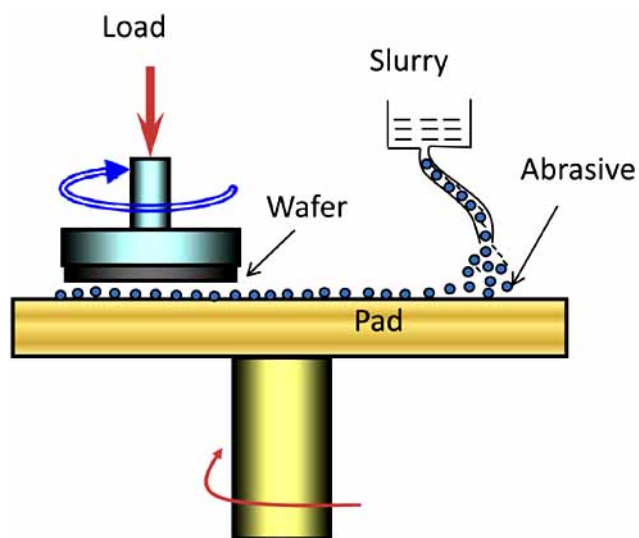
1. INTRODUCTION

Wafers of silicon, silicon carbide and sapphire are important materials in the semiconductor industry due to their thermal oxide property and stability [1-4]. However, the inherent brittleness and hardness of these wafer materials have limited their machinability under the rigorous requirements on surface and subsurface integrity [5-7].

The initial fabrication for the brittle wafers has been a mechanical polishing process that uses hard abrasives, such as diamond particles, to realise the material removal of the hard wafers by mechanical wear through the two-body and three-body contact sliding between the abrasives and the wafer. This process is easy to introduce a damaged subsurface layer (e.g., phase changes and dislocations). In order to improve the process, the chemo-mechanical polishing (CMP) technique has been introduced, in which certain chemical agents are used to aim to activate chemical reactions with the wafer material to facilitate the material removal and minimise subsurface damage. It has been claimed that CMP is one of the most powerful techniques to planarize brittle wafers, such as silicon, silicon carbide and sapphire [8, 9].

In a CMP process, a rotating wafer is pressed against a rotating polishing pad while slurry containing chemical agents and abrasive particles flows between the wafer and the pad, as illustrated in Fig. (**1**). The key in the development of an efficient CMP is to promote the combined chemical and mechanical interactions to maximise the material removal rate of the polishing and to minimise the possible damages to the wafer. Thus the efficiency of a CMP should be influenced by the chemical inertness of the wafer material to polish. For instance, it was reported that SiC and sapphire are more chemically inert than silicon, and hence it is more difficult to fabricate SiC and sapphire wafers by a CMP.

In the last decades, various CMP methods have been proposed to polish brittle wafers [10-13]. However, the process has been mostly trial-and-error owing to the lack of understanding of the material removal mechanisms. This has made the polishing process difficult to control.

This paper will review the CMP techniques used for the polishing of silicon, SiC and sapphire wafers, and provide some authors' comments on their merits, limitations and possible improvement.

Fig. (1). The schematic of a CMP process.

2. CMP OF SILICON

CMP of silicon wafers underpins the production of semiconductor devices, such as in the fabrication of integrated circuits (ICs). Due to the continuous demands for smaller device sizes and more complex circuit interconnection levels, it is required that the surface of each material layer is planarized upon pattern formation to its utmost, as

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otherwise it will be hard to form new satisfactory pattern layers over the top of the layer beneath. CMP-induced subsurface damage in silicon wafers is another point at stake, because fabrication damages incurring in an early stage of IC processing can be masked in the subsequent steps, leading to the failure of a device in its application, or can cause interruptive problems, such as wafer cracking, in the further fabrication phases. Therefore, planarization, damage-free subsurface generation and material removal efficiency are some of the important issues in the development of CMP.

2.1. Effect of Oxidizer

It was reported that oxidizer in slurry is very important to CMP, thus a popular attempt has been to vary oxidizers in slurry by using, e.g., H_2O_2 [14], NaOH, KOH, and Tetramethylammonium hydroxide (TMAH) [15], to improve a CMP process. On the other hand, Basim *et al.* [16] reported that salt in slurry, based on the ionic strength theory [17], could enhance the material removal rate in CMP. With the addition of NaCl, the removal rate could increase to 708 nm/min in conjunction with a surface roughness reduction to 0.86 nm. It was found that inorganic salts, such as KCl and $Ce(OH)_4$, could cause metal ion contamination in IC fabrication, and that the use of salts should be selective, e.g., using those consisting of tetramethyl ammonium hydroxide, tetraethyl ammoniumhydroxide, or silica/quaternary ammonium to reduce the metal ion contamination [18-20]. On the other hand, it was reported that the chemical reaction between wafer and slurry oxidizer was not significantly affected by the presence of potassium ions in water [21], and that amine-based additives can generally bring about a higher polishing rate, although the mechanism is unknown. Furthermore, the effect of salt on subsurface damage generation was not examined and unclear.

2.2. Effect of Abrasive Material

Different abrasive materials can alter CMP effectiveness. The most common abrasive for the CMP of silicon wafers is SiO_2 particles. Normally, the material removal rate is around 100 nm/min [14, 15, 22, 23]. To increase the rate, abrasives of higher hardness than silicon wafers have been tried [24, 25]. It was found that compared with silica and Al_2O_3 particles, ceria abrasives can result in higher polishing rates and fewer polish scratches on wafer surfaces. Thus, the application of the cerium oxide polishing slurry in polishing silicon wafers has been investigated [26], and some of such slurries have been commercialized [27]. Meanwhile, the organic compounds with an acetylene bond were added to further increase the polishing rate. Mixed slurries contain more than one type of abrasive materials [28, 29].

Abrasive geometry, e.g., sharpness, can influence polishing quality and cause damage to silicon wafers. Feng *et al.* [30] investigated a type of spherical and single-crystal nano-ceria abrasive particles of energetically unfavourable high index surfaces. They reported that the spherical nanoparticles largely reduced the surface defects in CMP process.

Two mechanisms were proposed to describe the higher material removal rate when using ceria abrasives. Cook *et al.* [31] and Osseo *et al.* [32], taking silica wafer as an example, considered that it was due to the chemical tooth mechanism

which caused the de-bonding of the temporary bonding between ceria abrasives and wafer substrate. However, a direct experimental validation is not available. Hoshino *et al.* [33], based on their observations, proposed that the removal of the wafer material by ceria abrasives was mechanical.

With the more and more stringent environmental protection demands, a green CMP technology is required. Estragnat *et al.* [25] studied the polishing of silicon wafer without using an oxidizer. The slurry contained only alumina particles, de-ionized water and pH additives. However, the material removal rate became lower (60 nm/min, at pH 12). Most recently, Biddut *et al.* [24] developed a chemical-free polishing technique for silicon wafers based on the stress-dependent phase transformation theory [34-38].

The performance of polishing pad influences significantly the polishing quality. Zhang *et al.* [39] studied experimentally the dependence of pad performance on its texture deterioration by investigating its correlation with polishing time, polishing pressure, and material removal rate. They concluded that material removal rate decreases as the cylindrical cell structure of a pad is gradually deteriorated, that there is a pad life limit beyond which polishing quality can no longer be maintained, and that the workable pad life can be extended to a certain degree by applying a higher polishing pressure. On the other hand, because of the deformation of a polymer pad, it is difficult to achieve a flat surface when using a polymer pad. Saito *et al.* [40] used a polish cloth made of ceramic material with silica abrasive to polish obtain flatter silicon wafers, but it is easier to cause surface scratch and subsurface damage.

Although some of the above CMP processes have been applied in semiconductor industry, the fundamental material removal mechanism is still vague, and some efforts have been placed to understand it considering abrasive indentation, scratching and ploughing [41], polishing conditions [42], effect of chemical factors on silicon's atomic bonding [43], or the effect of slurry properties [44, 45]. However, there are many open questions in the modelling.

3. CMP OF SILICON CARBIDE

SiC has a variety of electrical and thermo-physical properties that make it useful for semiconductor devices. These properties include high dielectric breakdown, high coefficient of thermal conductivity and high wear-resistant ability. However, there are some challenges for an efficient fabrication of SiC wafers. One is to produce a damage-free SiC wafer, while having a high material removal rate, because SiC is hard and chemically inert [46]. A conventional method for mirror finishing of SiC wafer is to polish the surface *via* hard abrasives such as diamond, SiC and Al_2O_3. However, damaged layers containing surface scratch and subsurface dislocation are difficult to avoid. CMP of SiC is both slow and expensive.

3.1. Additives in Slurry of SiC CMP

To enhance the polishing rate, White *et al.* [47] developed the CMP slurry formulations to polish single crystal SiC wafer while maintaining acceptable industry surface quality standards (roughness, scratches, etc.) It was

reported that a slurry without chemicals could provide a material removal rate of 2 nm/h, and a slurry without particles could give a polishing rate of 16 nm/h. The combined effect was a polishing rate of 0.2 µm/h with the obtained surface roughness of less than 0.4 nm. Since the slurry composition is commercially confidential, White *et al.* [47] only clarified a number of chemicals, pH and abrasives. Recently, Takemiya *et al.* [48] obtained a much higher removal rate using a polishing compound composed of some colloidal silica particles, an organic solvent, a nitric acid, a citric acid, H_2O_2 and a viscosity additive. The pH was around 12 buffered by KOH. To accelerate the polishing efficiency, diphosphorus pentaoxide was added to the slurry to reach the material removal rate of 6µm/h. However, the surface quality was not mentioned in this patent.

Minamihaba *et al.* [49] studied acid slurry to abrade the SiC surface with colloidal silica abrasives. The acid was selected from the group consisting of an amino acid and an organic acid having a benzene ring and a heterocycle, respectively. With optimizing the particle sizes (5 to 30 nm) and pH values (1 to 12), they claimed that if the primary particle size of colloidal silica exceeded 30 nm, it became difficult to remove SiC effectively. The influence of pH value on the material removal rate was shown in Fig. (**2**), indicating that the polishing slurry became incapable of material removal as pH fell within the neutral region. Preferable conditions would be in an acidic (pH =1~3) or an alkaline state (pH =8~11). At pH = 10, the polishing rate was the highest (3 µm/h). However, the surface quality was not addressed.
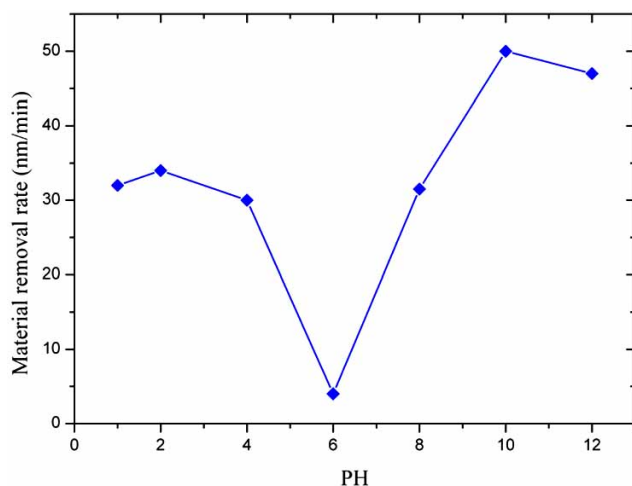

Fig. (2). Effect of pH values on the material removal rate in the CMP of SiC wafer [49].

Kerr *et al.* [50] provided slurry to produce a smooth, damage-free SiC wafer using ozonated water and hydrogen peroxide in a colloidal silica or alumina solution. It was considered that the dissolved ozone in the slurry was able to promote chemical reactions. The pH was buffered up to 8~14. To enhance the oxidation rate of SiC, the slurry temperature was increased, either by directly heating the slurry through chemical reactions. Kerr *et al.* [50] added an acidic or base solution in their polishing process, such as

H_2SO_4, KOH and NH_4OH, to stimulate an exothermic reaction to increase the temperature at the wafer surface. It was reported that by doing so the polished surface roughness reached 0.33 nm, but the removal rate was not presented. Matsui *et al.* [51, 52] applied oxygen gas or light to the polishing process to promote the oxidation rate of SiC with H_2O_2 as shown in Fig. (**3**), and claimed that this method could polish SiC at a low pressure. The surface roughness achieved was on the order of 0.5 nm. However, the material removal rate was not mentioned too. Generally, one may increase the material removal rate by using a higher polishing pressure or velocity, although this is usually limited by the capacity of an apparatus available and the higher possibility of subsurface damage.
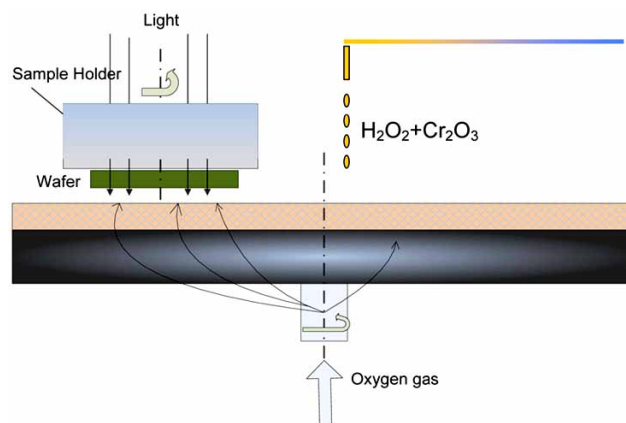

Fig. (3). Schematic of light and oxygen gas in CMP apparatus.

In summary, satisfactory slurry should be able to overcome the high activation barrier for breaking the Si-C bond. As proposed by White *et al.* [47], if the chemistry adsorbs to the particle surface at a saturated level, the maximum rate is possible to achieve. As illustrated in Fig. (**4**), the delivery of the chemistry directly to the Si-C bond *via* transport on the particle surface reduces the Si-C bond energy. The kinetic energy imparted by the particle momentum with the coulombic attraction force between the particle and wafer contributes to the high removal rate.
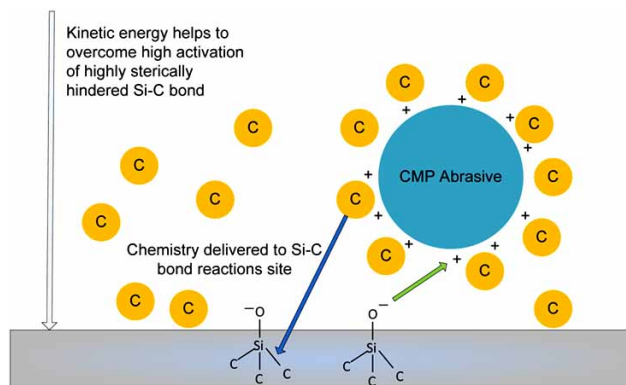

Fig. (4). The removal mechanism for SiC wafer in CMP slurry.

3.2. Step-Wise CMP

Li *et al.* [53] developed an electro-CMP (ECMP) technique by combining an anodic oxidation process with a normal CMP for the polishing of SiC, as illustrated in Fig. (**5**). The process consists of two steps. First, an anodic oxidation was executed using H_2O_2 for a short time. Secondly, the normal CMP process with silica abrasives at pH = 10 was carried out to removal the oxide. Since the colloidal silica abrasives are softer than SiC, the process only removes the oxide formed without introducing additional scratch. However, a high anodic current density could result in pitted surfaces. Thus the balance between anodic oxidation and oxide polishing was vital to realize a smooth and defect-free surface. Using this method, the surface roughness after polishing was around 0.27 nm, with a low material removal rate of 0.07 µm/h. Similar step-wise methods [54, 55] for the polishing of SiC wafers have been developed, using, for instance, diamond abrasives (<10µm) with hard polyurethane pads at a temperature around 70°C. The material removal rate achieved was 0.5~2µm/h. Then, to reduce surface scratches, SiO_2 abrasives (20~100nm) were subsequently applied to with softer polyurethane pads.

In short, by the means of the step-wise polishing, the obtained surface roughness of a wafer could be less than 1 nm, associated with a very low material removal rate (0.1~1µm/h). The method in general is costly.
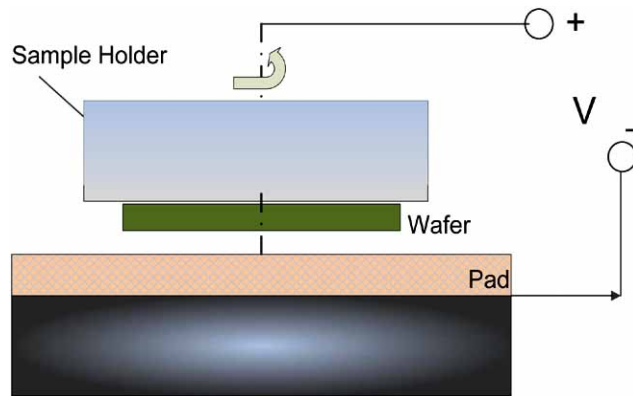

Fig. (5). Schematic of the ECMP setup.

3.3. CMP of SiC Using Mixed Abrasives

Polishing slurries containing mixed abrasives have become popular to improve the material removal rate and to minimise or eliminate damage. Following the traditional KOH-based colloidal silica CMP process of SiC wafer [56], An *et al.* [57] added NaOCl and diamond abrasives (25nm) to the conventional silica slurry. The pH value was 10.23. The polishing was carried out with the polishing pressure 80 KPa at the platen speed of 120 rpm. It was reported that the oxidizer (NaOCl) promoted the chemical reaction on SiC surface and led to the increase in the thickness of SiO_2 layer. The diamond particles added increased the polishing rate, reaching 0.1 µm/h. The surface roughness of the wafer was around 0.1 nm.

3.4. CMP of SiC Without Abrasives

To avoid involving abrasives, Lin *et al.* [58] used a metal disc to replace a polymeric pad to polish a SiC wafer in the slurry with water and kerosene. The experimental results demonstrated that using a cast iron or a stainless steel disc could produce a better surface quality than that of using a copper disc. The hardness of a polishing disc was not a dominant factor. They speculated that the iron oxide formed when using ferrous metal discs might reduce the activation energy and thus the threshold temperature of the tribo-chemical reaction between SiC and water. Although this method could avoid chemical pollution, the material removal rate was low, at about 0.06 µm/h, and the surface roughness obtained was high, at about 20 nm. The polishing of SiC wafers using a metal disc is similar to the dynamic friction polishing of diamond and diamond composites [59-61].

4. CMP OF SAPPHIRE

Sapphire is widely used in a range of electronic and optical parts, viewports, sight windows, substrates and semiconductor wafers due to its excellent optical, mechanical and chemical properties. For example, sapphire retains its high strength at high temperatures, and has a good resistance to scratching, stable thermal properties and excellent chemical stability. In many of the above applications, a sapphire wafer needs a flat, super-smooth and free-damage subsurface. However, effective CMP of sapphire is still unavailable [62, 63]. This section reviews some of the current methods.

4.1. Chemical- or Mechanical- Polishing Method for Sapphire Wafer

In general, to polish sapphire chemically requires a higher temperature (above 200°C) [64, 65], but to polish it mechanically is easy to introduce subsurface damage [66]. Dislocation pairs could be periodically formed along the region parallel to a polished surface.

4.2. Slurry for the CMP of Sapphire

Moeggenborg *et al.* [67] fabricated a sapphire surface in CMP using colloidal silica abrasive particles. The pH value of the CMO slurry was 10~11, adjusted by KOH. Inorganic salt compound, such as NaCl, KI and NaI, was added into the aqueous medium solution. The polishing experiment was carried out on a Logitech CDP machine with the pressure of 79.35KPa at the platen speed of 65 rpm. The removal rate of sapphire was 5.28 µm/h. However, without the salt, the removal rate was reduced to 2.7 µm/h. The surface quality was not mentioned in this patent. Cherian *et al.* [68] also applied the salt-additive to the slurry to promote material removal rate. Zhu *et al.* [69] used alumina slurry. The experiment was conducted on a precision polishmaster (Stras-Baugh) at the plate speed of 60 rpm with an IC1000 pad (Rodel). The effects of the pH value and pressure were optimized. It was noted that the highest removal rate was approximately 0.89µm/h with a RMS = 0.3 nm in a 20×20 µm area. It was believed that a coupling effect of the chemo-mechanical reaction between the sapphire and alumina accelerated the material removal. However, the performance

in terms of material removal rate still needs to be further improved. Wang *et al.* [70] achieved a RMS of 0.23 nm for sapphire wafer by a step-wise technique (CMP plus a subsequent chemical etching); but the removal rate was not mentioned. Sun [71] obtained a destructive removal rate of 7.25μm/h with the pressure of 13.78 KPa and platen speed of 50 rpm at pH = 11. The conductivity of the silica slurry was adjusted by pH value and complex chemicals, such as EDTA. However, the surface quality was not reported.

To enhance the chemical reaction between sapphire and slurry, Liu *et al.* [72] provided a chemical solution composed of a chelating agent, which has 13 EDTA chelate rings such as a mixture of EDTA and KOH, and a strong propensity for complexation with aluminium ions and for forming a water-soluble chelating product. To avoid the damage from the hard abrasives, silica abrasives were used. The experiment result showed that the removal rate was 9.96~18μm/h and the surface roughness was 0.1~0.3 nm. However, the details of the polishing parameters, such as pressure and plate speed, were not mentioned in this patent.

4.3. CMP of Sapphire Using Mixed and Coated Abrasives

Hu *et al.* [73] polished sapphire wafers using micrometer B_4C and nanometer silica abrasives. The pH value was 12, adjusted by NaOH. It was observed that the removal rate was 2.27μm/h at the polishing pressure of 55 KPa and plate speed of 60 rpm, which is comparable to the others published in the literature (e.g., removal rate = 2.4μm/h, pressure = 80 KPa, plate speed = 65 rpm and pH = 10 [11]). The surfaces before and after polishing were characterized by XRD, as shown in Fig. (**6**). The full width at half maximum (FWHM) of diffraction peak reduced from 63.8s before polishing to 21.2s after polishing, indicating that the surface quality was remarkably improved. Meanwhile, the RMS was reduced from 99.36 nm before polishing to 0.71 nm after polishing.
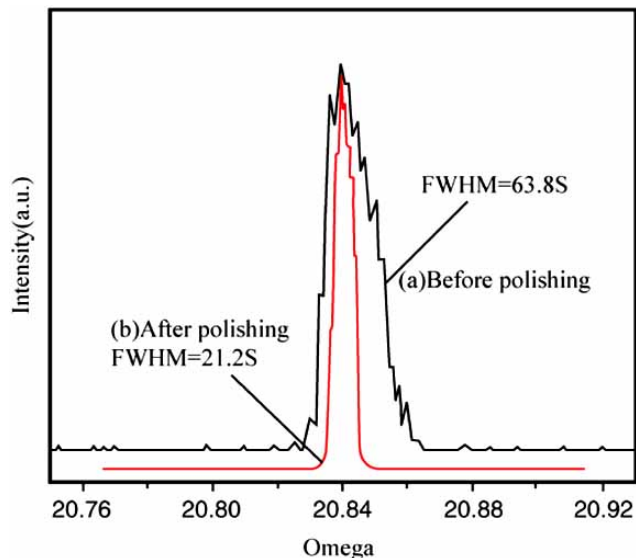

Fig. (6). XRD curves of sapphire substrate (**a**): before and (**b**) after polishing [73].

Bakshi *et al.* [74-76] developed a slurry mixture composed of silicon carbide and alumina particles with an aqueous medium. When using a platen speed of 400 rpm, the material removal rate reached 11.4μm/h. The surface roughness was sharply reduced after 60 minutes, as shown in Fig. (**7**). It was thought that the surfaces of the SiC abrasives were slightly oxidized and, thus, reacted with the sapphire surface to facilitate the material removal. However, since both of the SiC and Al_2O_3 abrasives are hard particles, it is likely that the hardness of the particle promoted the polishing rate of the sapphire wafer. This seems to be aligned with the higher surface roughness (~1nm) after polishing [69, 70, 72].
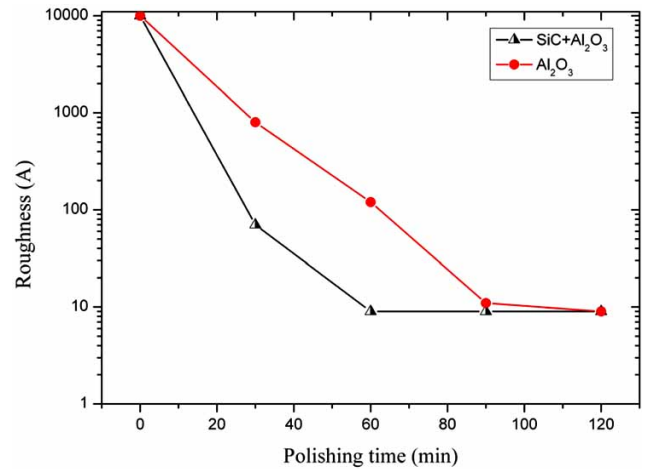

Fig. (7). The relationship between the surface roughness and polishing time [74].

Apart from the simple mixture of two-types of abrasives, coated abrasives (CAs) were recently proposed for the CMP of sapphire. CAs are synthesized by the following process: (i) adding certain amount of micro hard abrasives, such as SiC, to deionized water, followed by an ultrasound treatment; (ii) adding certain nano soft abrasives, such as silica and ceria, to the suspension obtained in step (i), followed by an ultrasound treatment again; and (iii) adjusting the slurry by chemicals or pH value. Wang *et al.* [77] used the coated abrasives, such as B_4C and SiO_2, B_4C and $α-Al_2O_3$, B_4C and CeO_2, to polish sapphire wafers to improve the removal rate and surface quality. The experiments were designed on a CMP tester (Cetr CP-4) with the pressure of 70 KPa at the plate speed of 150 rpm. The removal rates of the above three coated-abrasives were 6 μm/h, 10.8 μm/h and 9 μm/h, respectively. Meanwhile, the RMS of the wafer surface after polishing was less than 0.75 nm. They also presented a schematic mechanism for using the CAs, as illustrated in Fig. (**8**). Compared to the size of ceria and boron carbide particles, the distance of their interaction in light of chemical bonds and electrostatic force could be ignored. In addition, since the size of boron carbide is much larger than that of ceria, it seems that the ceria particles rightly seats on the surface of boron carbide core. It can be considered that, with the gradual increase in ceria particle on the core particle surface, the interaction between particles becomes more and more complete, leading to a more adequate chemical reaction between the outer ceria particles of the CAs and the sapphire
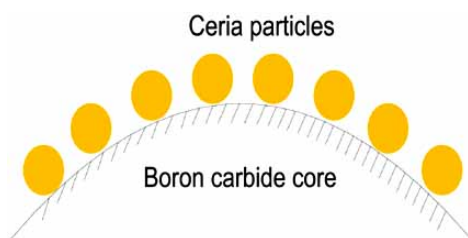
Fig. (8). The schematic removal mechanism for the CA particle in sapphire CMP [77].

hydration layer formed in CMP. Meanwhile, a severe mechanical tear appeared when using the hard B_4C core. Due to the dualistic function with respect to the direct contact between a soft particle and the sapphire hydration layer and a fierce indirect mechanical removal of hard particle, desired performance in terms of high removal rate and surface quality can be reached.

5. CURRENT & FUTURE DEVELOPMENTS

This paper reviews some of the present status of CMP for brittle wafers including silicon, SiC and sapphire. It is clear that in all the methods discussed, two main sets of factors are involved to improve the polishing rate and surface quality.

The first set of the factors is mechanical, such as polishing pressure, speed, abrasive hardness and size. They influence polishing rates. A high polishing pressure and speed will result in a high material removal rate but will also increase the possibility of surface scratching and subsurface damage. To minimise such negative effects, it is essential to understand the forces on a single abrasive particle so that possible optimization can become possible. The second set of the factors is chemical. Many inorganic and organic chemicals, such as oxidizers (i.e. H_2O_2, NaOCl, KOH, TMAH), salts (i.e. NaCl, KI, EDTA), have been used in polishing slurries to enhance the oxidizing rate of wafer. Furthermore, light, heat and oxygen gas have also been used to accelerate the chemical reaction, especially in the case of polishing SiC and sapphire wafers. However, the improvement by adding chemicals in polishing SiC and sapphire wafers are not remarkable. On the other hand, the usage of chemicals leads to pollution.

Damage-free polishing without using any chemicals has been successfully demonstrated in polishing silicon wafers [24]. The mechanism is to make use of the re-crystallisation of amorphous silicon before the emergence of β-tin [37]. If one wants to achieve damage-free polishing without chemicals for SiC or sapphire wafers, methods based on different mechanisms must be established. Fundamental investigations into the mechanisms of material removal and subsurface damage [78-81] will be particularly useful.

As can be seen clearly, the CMP of brittle wafers is a very complicated process involving many factors that cannot be readily modelled and precisely controlled, such the shapes and sizes of abrasives, the properties and conditions of polishing pads, and even the polishing process parameters, e.g., real contacting forces – to date there is not a method or a model which is able to describe accurately the interaction

force/stress between a wafer and an abrasive particle [39]. These call for further extensive studies.

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CONFLICT OF INTEREST

The authors have no conflict of interest with any references listed in this paper.

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