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chemical-mechanical slurry, polishing,
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Le Anh DUC[1*], Pham Minh HIEU[2*],
Nguyen Trong MAI[3], Thai Van TRONG[3],
Nguyen Minh QUANG[3]

ANALYSIS OF SOLID AND IONIC SURFACE REACTION FORM TO SURFACE QUALITY WHEN USING CHEMICAL-MECHANICAL SLURRY POLISHING

The process of removing machining residues using chemical-mechanical slurry (CMS) has an important place in the creation of ultra-precise components in optical devices. Based on this feature, this work investigates the efficiency of the CMS polishing process by comparing the surface reaction modes by the ionic and solid reaction modes when polishing the yttrium aluminum garnet (YAG) and sapphire crystal. The study procedures were conducted to clarify the polishing performance corresponding to these two reaction types. The obtained experiments results show that the balance between the mechanical effect process using CMS polishing technology with chemical effect can be achieved with the ionic reaction mode. The results also show that the ionic surface reaction modes give more uniform material removal than the solid reaction on YAG and sapphire crystal surfaces. Therefore, the surface quality when polished by CMS technology with ionic surface reaction modes is better than that of solid surface reaction.

1. INTRODUCTION

The workability and performance of optical elements are greatly influenced by their surface quality. In which optical devices have improved performance and quality when surface smoothness is improved [1, 2]. Under the combined action of mechanical force components with chemical reactions in chemical mechanical slurry (CMS) polishing processes, this process enables the ability to remove excess material in nano form and along with which superfine surfaces are generated [3, 4]. In the ultra-precision machining processes of optical devices, studies by Wang et al. [5] have shown that the polishing method using CMS is one of the effective methods used.

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[1] Faculty of Information Technology, Hanoi University of Industry, Hanoi city, Vietnam
[2] Faculty of Automobile Technology, Hanoi University of Industry, Hanoi city, Vietnam
[3] Faculty of Mechanical Engineering, Hanoi University of Industry, Hanoi city, Vietnam
[*] E-mail: ducla@haui.edu.vn, hieupm@haui.edu.vn

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There are two main reactions in the CMS polishing process that is ionic and solid format. When polishing with colloidal silica slurries, the reaction in solid form is created between SiO_2 abrasive particles in colloidal slurries with workpiece surfaces, then a soft surface layer will be formed. SiO_2 abrasive particles in the colloidal slurry are used for the polishing processes as shown in Fig. 1. In slurry form, silica abrasive particles exist as structure –Si–O–Si– combined with –OH to form the –Si–OH bond [6, 7]. By dehydration, the –Si–OH components in the polishing process with colloidal silica slurry will react with W atoms in the wafer surface will create a connection Si–O–W [8, 9]. Under the influence of mechanical movements produced by the polishing process, the ability to remove materials is described in Fig. 2.

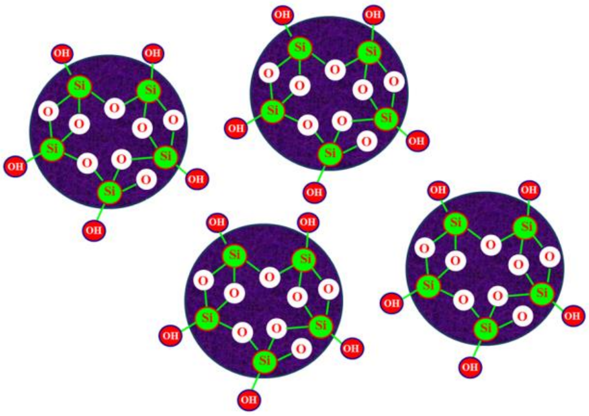


Fig. 1. Si–OH bond formation process in silica slurry [6]

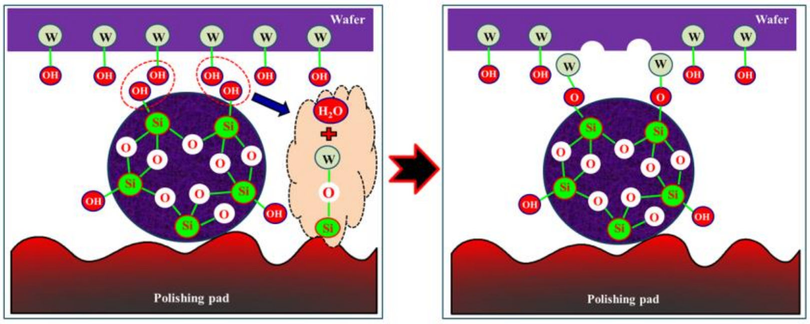


Fig. 2. Principle of remove excess material by CMS polishing process using silica slurry [9]

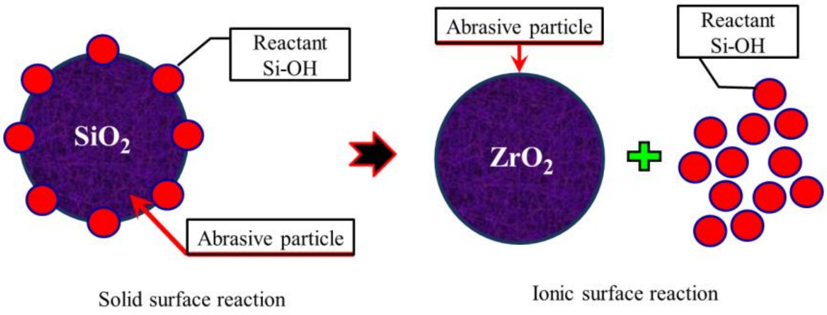


Fig. 3. Solid and ionic reaction transformation process in CMS polishing [10]

The reaction modes between workpiece surfaces and CMS can be created in ion form. By the way, the ionic reaction modes can be realized using Sodium Metasilicate Pentahydrate (Na_2SiO_3–$5H_2O$) in the CMS [10]. The reactions are created in ionic form instead of solid as described in Fig. 3. Aim to dissect reaction mode's effect to polishing process of YAG and sapphire crystals by CMS, in this work, two reaction modes of solid and ionic surfaces in the same chemical reaction were analyzed. After polishing by CMSs method, the ability to remove materials along with surface morphology is determined through XPS and atomic force microscopes. Results obtained when applied to the CMS polishing processes show that the ionic surface reaction modes have a balanced combination of mechanical and chemical effects, thereby giving the ability to remove material excess more uniformly than in solid surface reaction modes. Hence the better polishing quality is obtained by ionic surface reactions.

2. EXPERIMENTAL DETAIL AND METHODOLOGY

Two types of CMS used in polishing were produced to obtain two surface reaction modes described in Table 1. Solid surface reaction modes were generated from a silica slurry containing 9% by mass of SiO_2 abrasive particles with a diameter of 50 nm. The ionic surface reaction modes are produced by a mixture of ZrO_2 and Na_2SiO_3–$5H_2O$. Aim to find the most suitable conditions to create ultra-fine surfaces in polishing processes by CMS, the concentration of the Na_2SiO_3–$5H_2O$ reactant was determined according to the YAG crystal polishing experiments [11, 12]. When the concentration of Na_2SiO_3–$5H_2O$ increases, the chemical effects increase, and according to Zhang [13] study showed that the mass ratio of Na_2SiO_3–$5H_2O$ in CMS is 5%, which will have a balanced chemical and mechanical effect in the polishing process, along with the best surface quality obtained from the workpiece. In order to exclude objective factors affecting the CMS polishing process, in the polishing processes the size of the abrasive particles and their mass percentage did not change in the experimental processes. Polishing processes using CMS were performed on YAG and sapphire crystals, thereby evaluating the reaction modes to the machined surface quality. The hardness of the workpieces and abrasive particles materials according to Moh standard is listed in Table 2 [14]. The pre-polished wafers are ground on a 3M diamond grit plate. The wafer grinding and polishing processes are performed on the Smart-LAM 3.0 polishing machine. The polishing processes with CMS-1 and CMS-2 for the YAG and sapphire crystals workpiece are carried out for 90 min. The CMS polishing process parameters are described in Table 3.

Table 1. CMS polishing components

No.	Abrasive particles	Abrasive particle size (nm)	Percentage of abrasive particles (%)	Percentage of Na_2SiO_3–$5H_2O$ (%)
CMS-1	SiO_2	50	9	–
CMS-2	ZrO_2	50	9	5

Table 2. The hardness of abrasive particles and polishing materials according to Moh standard

Material	ZrO_2	SiO_2	YAG	Sapphire
Hardness	7.5	7	8.5	9

Table 3. Machining parameters when grinding and polishing on the Smart-LAM 3.0 machine

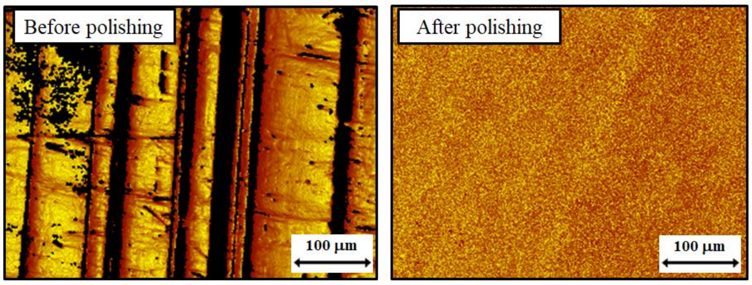
Technological process	Pad composition	Rotation speed (rpm)	Pressure (MPa)	CMS flow speed (ml/min)
Grinding	673LA–3M	60	0.05	8
Polishing	Polyurethane IC1000	90	0.05	8

Surface morphology was observed by optical microscopes GX-51 Olympus and AFM-XE-200. The material remove rate (MRR) was determined through the scraping method. ESCALAB-250X instrument used in XPS analysis. Surface roughness is measured on a Zygo NewView 6100 surface roughness test.

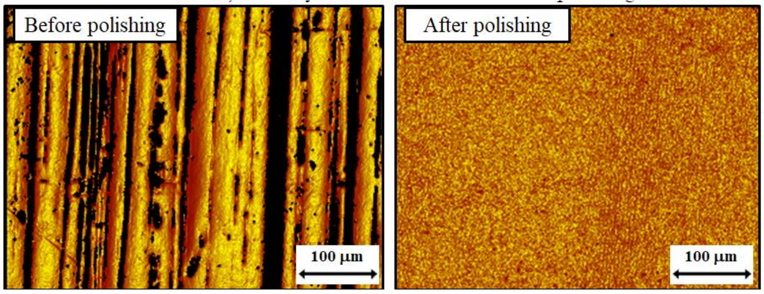
3. RESULTS AND DISCUSSION

3.1. CMS POLISHING PROCESS WITH DIFFERENT MATERIALS

Aim to investigate the polishing ability of CMS-2 with different materials, the polishing processes were performed on YAG and sapphire crystals, the surface image results obtained before and after polishing as described in Fig. 4.



a) YAG crystal surface before and after polishing



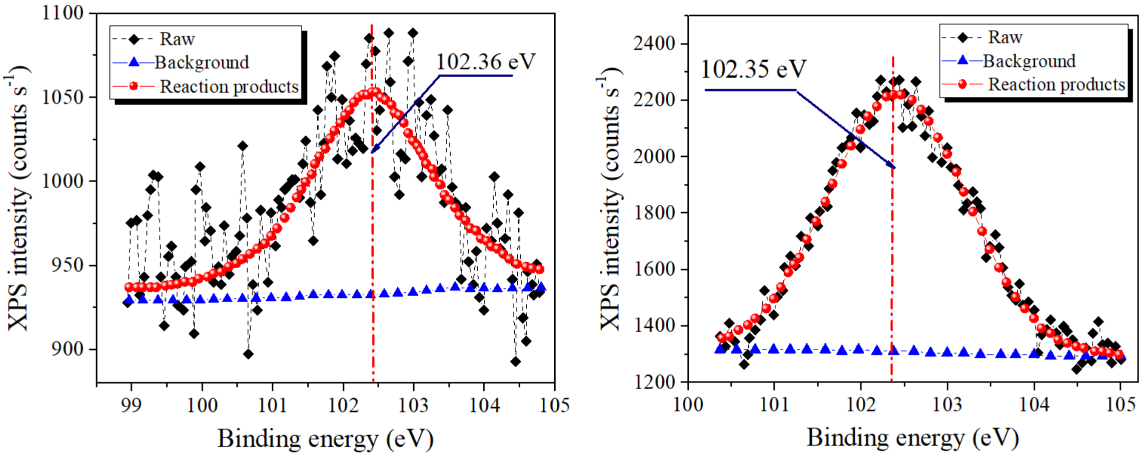
b) Sopohire crystal surface before and after polishing

Fig. 4. SEM images of different workpiece materials when polishing by CMS-2

The pre-polished surfaces have many scratches created by the grinding process on the surface of the workpiece. The polishing results show that the chemical effect of Na_2SiO_3–$5H_2O$ in CMS-2 plays an important role in polishing sapphire and YAG crystals because these crystals have a higher hardness than ZrO_2 abrasive particles, therefore, the mere mechanical impact on material removal is negligible. Because Na_2SiO_3–$5H_2O$ reacts with the elements on the surface of the workpiece to create a new layer of material with hardness not equal to the ZrO_2 abrasive particles, so under the mechanical impact of the polishing process this material layer is easily taken away.

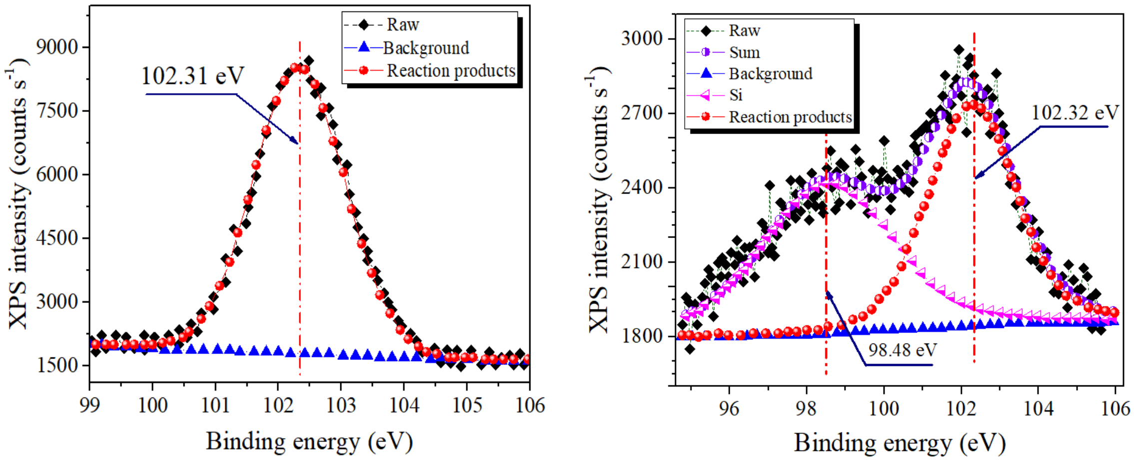
3.2. CHEMICAL REACTIONS WHEN POLISHING WITH CMS

When applying the CMS method in polishing YAG and sapphire crystal surfaces, XPS analysis processes were performed to investigate the surface layer chemical reactions when polishing by CMS-1 and CMS-2. The results of XPS analysis show that the Si 2p spectrum after polishing the YAG crystals by CMS-1 and CMS-2 is depicted in Fig. 5, image shows that the reaction layers with the two types of CMS have the same characteristics. The results when polishing sapphire crystals are similar to polishing YAG crystals, these reactive layers also have the same characteristics. After polishing by CMS-1 the Si 2p peak (102.36 eV) on the YAG crystal surface, with CMS-2 the Si 2p peak (102.35), both polishing methods are consistent with them to produce a mixture of Y–O–Si and Al–O–Si. Figure 6 shows Si 2p peak in the workpiece surface after polishing sapphire crystal with CMS-1 (102.31 eV) and CMS-2 (102.32 eV and 98.48 eV). It can be seen that the surface after polishing with CMS-1 exists chemical bonds Al–O–Si, while CMS-2 besides Al–O–Si still exists Si. The analysis results of Si 2p bond energy of two polishing methods both show the same, thereby, it shows that the polishing process of the same material (YAG or sapphire) with CMS-1 and CMS-2 is the same. In addition, the Si 2p peak parameters of different bonds in polishing with CMS are listed in Table 4 [15, 16].



a) After polishing by CMS-1 b) After polishing by CMS-2

Fig. 5. Si 2p spectrum after polishing by CMS-1 and CMS-2 of YAG crystals



a) After polishing by CMS-1 b) After polishing by CMS-2

Fig. 6. Si 2p spectrum after polishing by CMS-1 and CMS-2 of sapphire crystals

Table 4. Si 2p binding energy

Material	SiO_2	Si	Na_2SiO_3
Binding energy (eV)	103.6	98.4	101.8

In the polishing processes by CMS-1, the hard surface reaction occurs between the –Si–OH groups that exist on the SiO_2 abrasive surface and the workpiece surface. Meanwhile, polishing by CMS-2, Na_2SiO_3–$5H_2O$ present in the slurry produces SiO_3^{2-}, due to hydrolysis when the concentration of Na_2SiO_3–$5H_2O$ is low, SiO_3^{2-} forms exist mainly in the structure $HSiO_4^{3-}$, $H_2SiO_4^{2-}$ and $H_3SiO_4^{-}$, these ions both contain the –Si–OH structure. Therefore, the ionic surface reactions occurring when polishing by CMS-2 are generated by the –Si–OH groups in a solution that exist as ions and the workpiece surface.

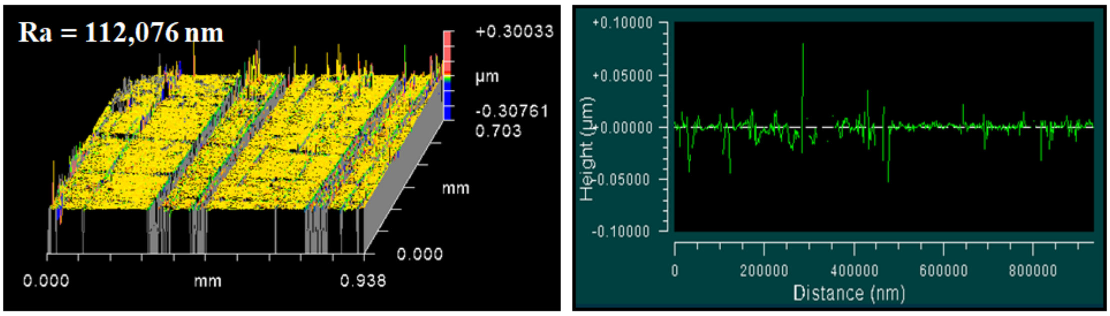
The above analysis shows that different forms of –Si–OH are generated by the polishing process by CMS-1 and CMS-2 and together produce a reaction product on the same workpiece surface. With the polishing process by CMS-1 for hard surface reaction, CMS-2 for the ionic reaction [17] with the reaction mechanism shown by:

$$\text{Si–O–H} + \text{H–O–W} \rightarrow \text{Si–O–W} + H_2O$$

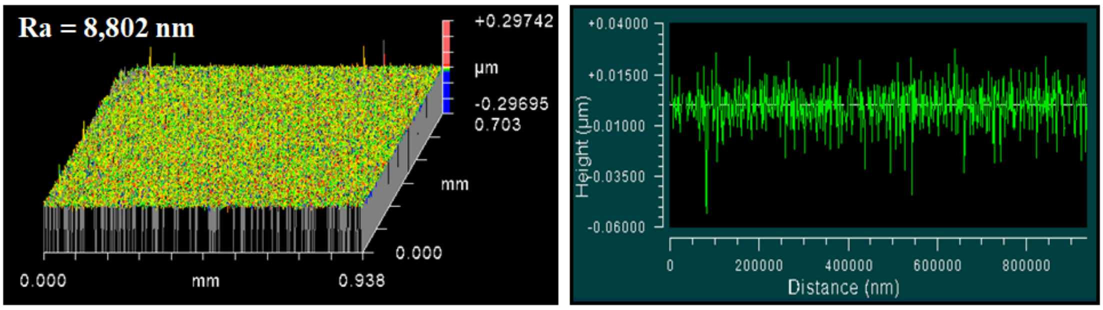
3.3. PERFORMANCE AND ABILITY TO REMOVE EXCESS MATERIAL UNIFORM WHEN POLISHING

3.3.1. POLISHED PERFORMANCE BY CMS TECHNOLOGY

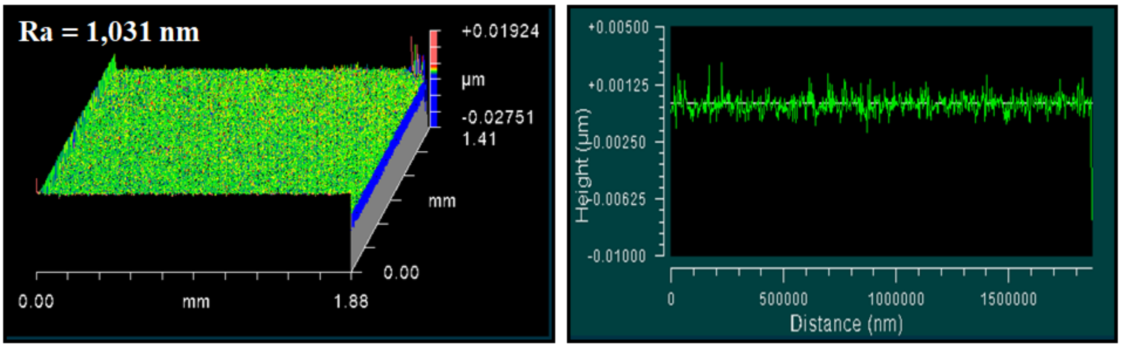
The polishing processes performed by CMS-1 and CMS-2 are applied to the YAG crystal and the sapphire in order to better compare the performance between them. The obtained results show that both polishing methods are effective on different workpieces, along with the same surface layer chemical reactions produced by different CMSs, but the reactions modes are different. The surface quality of YAG and sapphire crystal after polishing by CMS-1 and CMS-2 is shown in Fig. 7 and 8. Along with that, the ability to material remove rate and surface roughness parameter according to Ra after polishing is also achieved shown in Fig. 9.



a) Surface morphology of YAG crystal before polishing



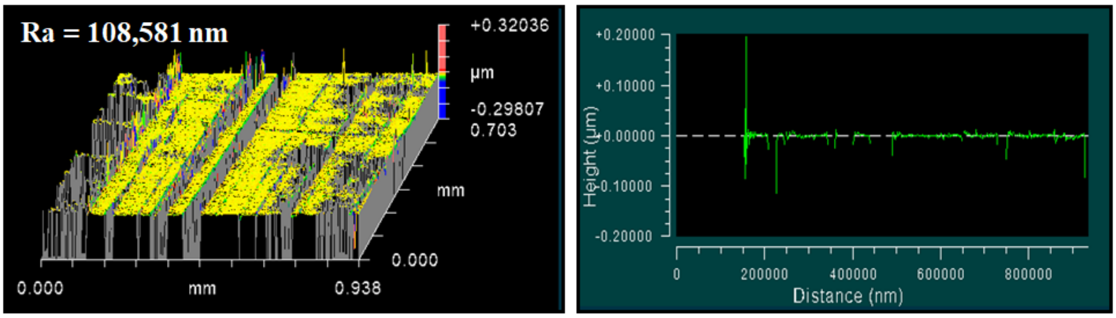
b) Surface morphology of YAG after polishing by CMS-1



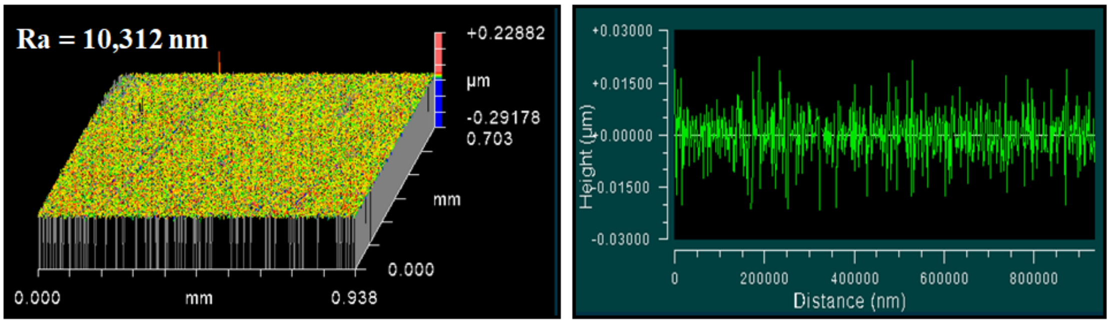
c) Surface morphology of YAG after polishing by CMS-2

Fig. 7. Surface morphology of YAG crystal before and after polishing by CMS-1 and CMS-2

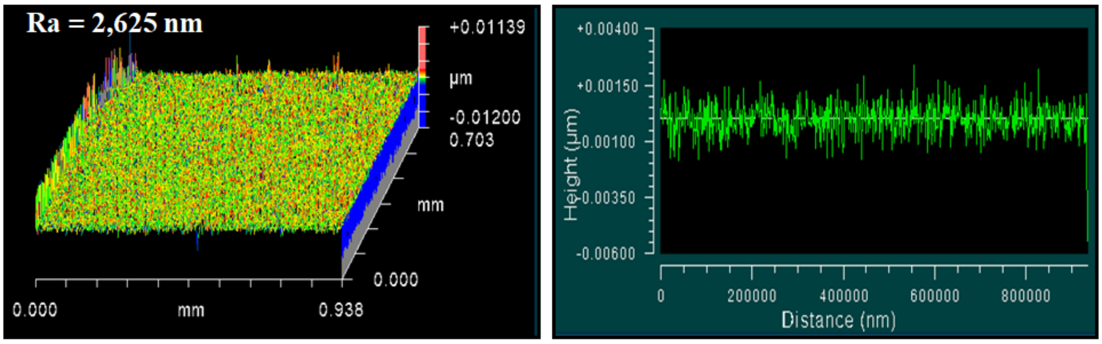
Before polishing the workpieces of sapphire and YAG crystals are mechanically ground with a surface roughness of about Ra = 110 nm. After polishing by CMS-1, surface roughness was obtained with the sapphire crystal workpiece (10.312 nm), with YAG crystal workpiece YAG (Ra = 8.802 nm). When polishing by CMS-2 the surface quality is improved compared to CMS-1, with Ra being 1.031 nm for YAG crystals and 2.625 nm for sapphire crystals, respectively. Compared with the effect of CSM-1, the polishing ability by CSM-2 gives a significantly improved surface quality. The cause of this phenomenon is created by the difference in the reaction surface modes of the surface layer and will be analyzed more clearly by the authors in the following section.



a) Surface before polishing

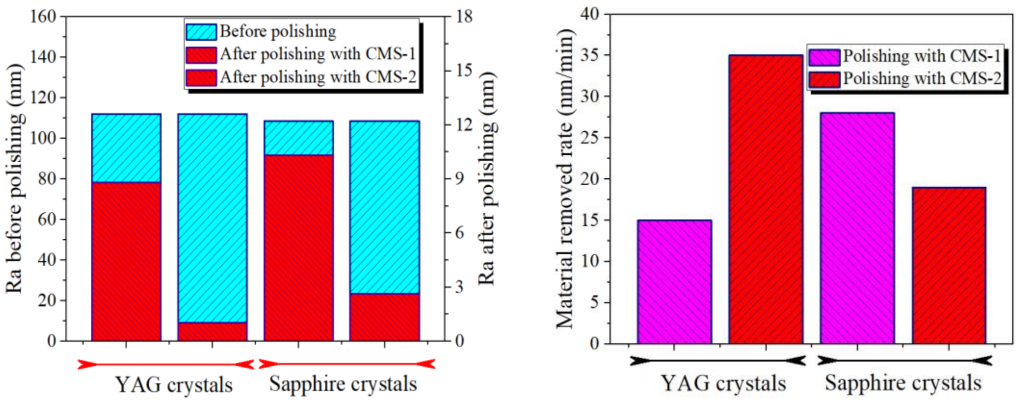


b) Surface after polishing by CMS-1



c) Surface after polishing by CMS-2

Fig. 8. Surface morphology before and after when polishing sapphire crystal by CMS-1 and CMS-2



a) Surface roughness b) Material removed rate

Fig. 9. The polishing ability with CMS-1 and CMS-2

3.3.2. THE MECHANICAL AND CHEMICAL BALANCE IN POLISHING BY CMS

Aim to investigate the polishing ability of YAG crystal workpiece by CMS-1 and CMS-2, the surface morphology was investigated at different locations in surface workpiece as depicted in Fig. 10.

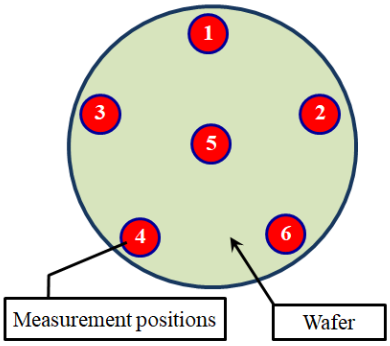
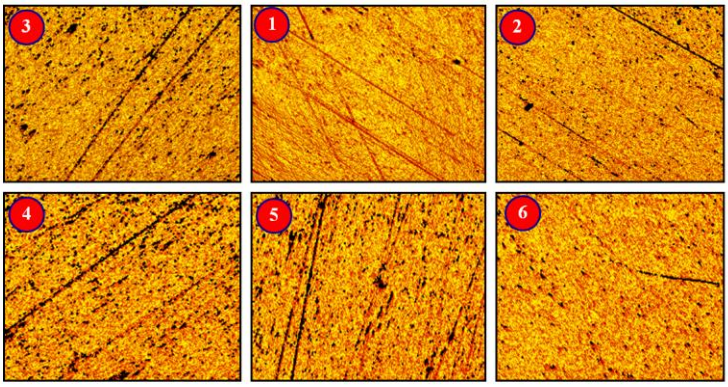
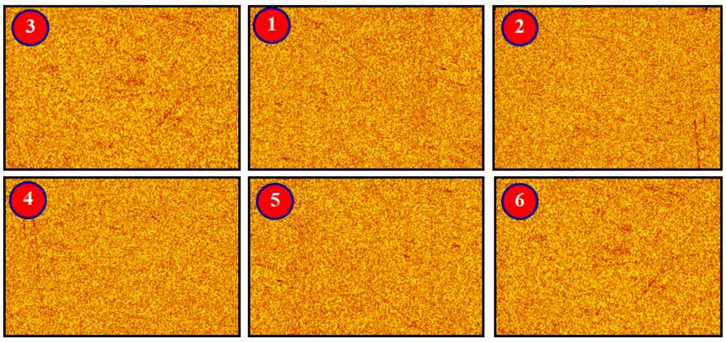


Fig. 10. Surface morphology analysis points

The cross-sectional scratches are depicted in Fig. 11. The results showed that many small scratches remained on the surface after polishing by CMS-1, whereas when polishing with CMS-2 an ultra-smooth surface was created and almost no scratches were observed at different viewing positions on the surface of the post-machined.

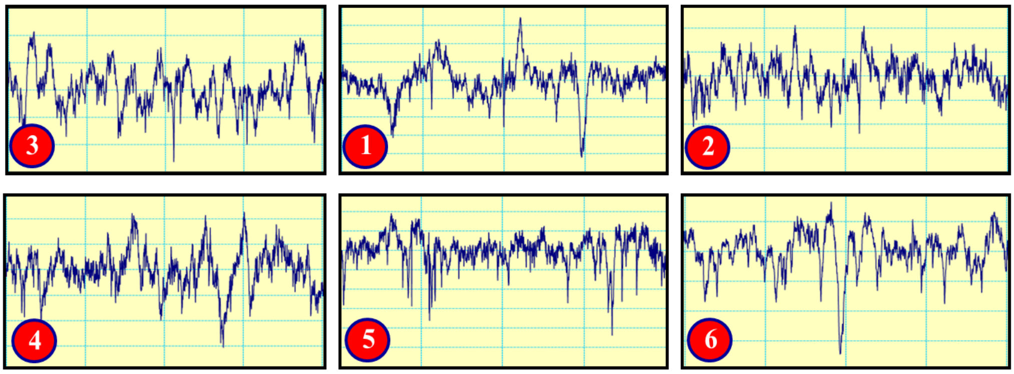


a) Polishing by CMS-1

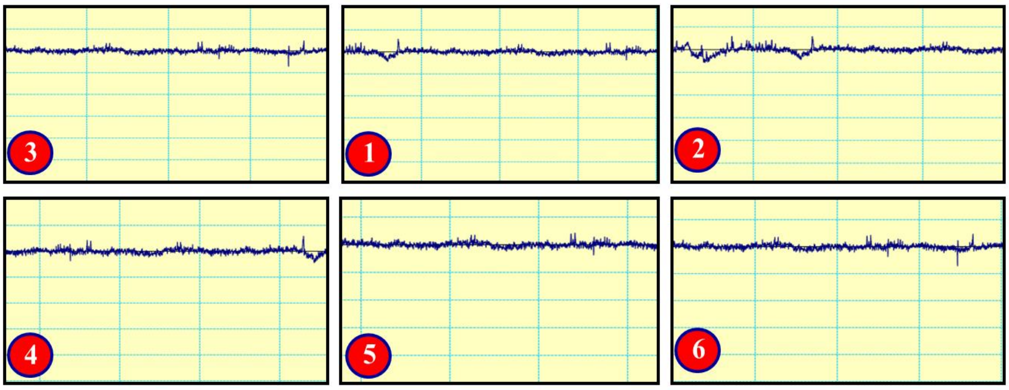


b) Polishing by CMS-2

Fig. 11. SEM images of YAG crystals at different locations after polishing by CMS-1 and CMS-2



a) Cross-section with CMS-1



b) Cross-section with CMS-2

Fig. 12. Cross-section at different positions after polishing by CMS-1 and CMS-2 of YAG crystals

The analysis results based on surface morphology show that polishing by CMS-2 gives a significant improvement in surface quality compared to polishing by CMS-1. Thereby, to achieve a super smooth surface during polishing by CMS it is necessary to balance mechanical and chemical effects [18]. The description in Fig. 13 shows that with the polishing process by CMS-1 when the surface of the YAG workpiece is in contact with the SiO_2 abrasive, it will react with the –Si–OH group on the surface layer. Due to the solid reaction by the contact process therefor the content of -Si-OH is difficult to adjust during polishing with CMS-1, making it difficult to maintain chemical and mechanical balance when polishing. It is this feature that creates a polished surface with small scratches due to the strong mechanical force that occurs after polishing by CMS-1 [19].

When polishing with CMS-2, the –Si–OH group is formed as ionic and not associated with the abrasive as depicted in Fig. 14. With this feature, the mechanical and chemical effects in the CMS polishing process will be modified by changing the number of abrasive particles and the concentration of $Na2SiO_3$–$5H_2O$. In the machining process, if the chemical reaction is weak, it is necessary to add more Na2SiO3-5H2O content to the CMS, which will facilitate the –Si–OH components to be more distributed on the surface of the workpiece, thereby increasing improves reaction speed and correcting the balance between mechanical and chemical in polishing by CMS. This balance action results in an ultra-smooth YAG crystal surface without scratches, with a roughness obtained in the nanoscale (Ra = 1 nm) after polishing by CMS-2.

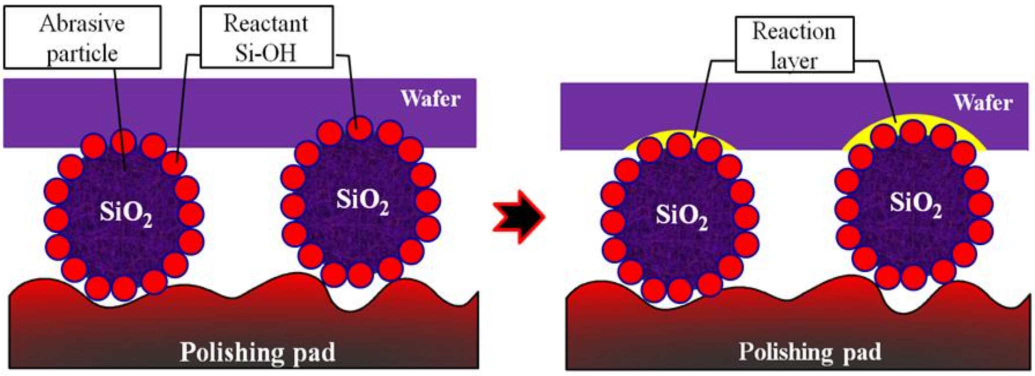


Fig. 13. Mechanical and chemical action in CMS-1

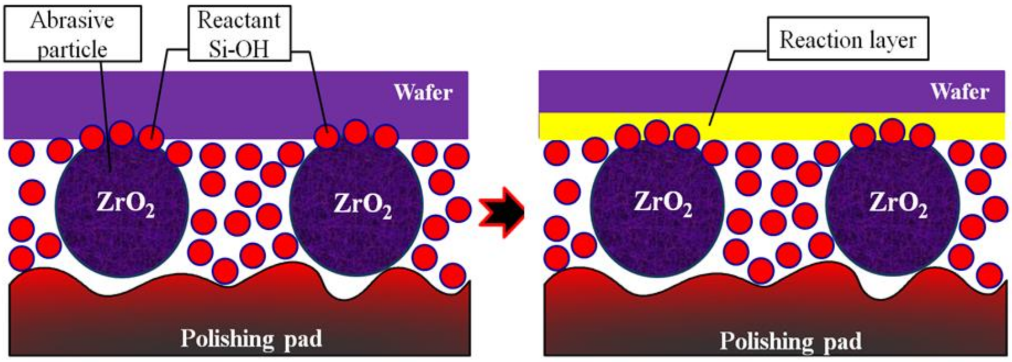


Fig. 14. Mechanical and chemical action in CMS-2

3.3.3. ABILITY TO REMOVE MATERIAL EXCESS EVENLY

Uniformity in the removal of workpiece residues is a very important factor for the creation of ultra-smooth surfaces [20]. Because of this property, the authors investigated differences in the ability to remove uniform residues in polishing processes by CMS-1 and CMS-2. Where the ability to remove workpiece residues by CMS polishing processes depending on the surface reaction layer. The surface reaction layer depth is highly dependent –Si–OH groups, with a higher concentration of –Si–OH giving rise to a larger reactive surface layer depth. The polishing process of the workpiece by CMS-1 gives a non-uniform reaction layer depth as depicted in Fig. 15. This process occurs because the size of the SiO_2 abrasive particles affects the chemical activity of –Si–OH in CMS-1. Smaller SiO_2 abrasive particles will provide higher speed and activity in CMS polishing. During the polishing process with CMS–1, SiO_2 abrasive particles have –Si–OH structures, however, this structure will decrease when reacting with the workpiece surface [21, 22]. In polishing processes by CMS-1, abrasives with more –Si–OH groups on the surface layer will create more Si–O–W bonds with the workpiece surface [23]. From the above characteristics, when polishing by CMS-1, the uneven chemical reactions on the surface lead to uneven soft layer depth, so the process of removing the uneven machining residue on the surface wafer and high roughness is obtained.

During polishing by CMS-2, the reaction –Si–OH and wafer surface is an ionic (liquid) reaction, on the wafer surface there is a lot of –Si–OH in the ionic form. This process created chemical reactions that take place evenly and unaffected number of –Si–OH groups on the abrasive particles and the abrasive particle size, with the action of the proposed ionic reaction, a very thin molecular-scale soft reactive layer on the wafer surface is generated, is uniformly generated on the surface and the abrasive particles easily remove the entire soft reactive layer as shown in Fig. 16. This process allows removing the surface layer of the workpiece uniformly, thereby improving the surface quality.

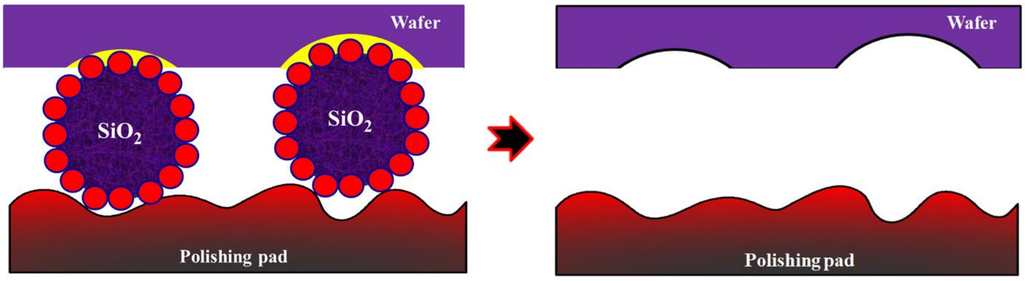


Fig. 15. Material removal capacity when polishing by CMS-1

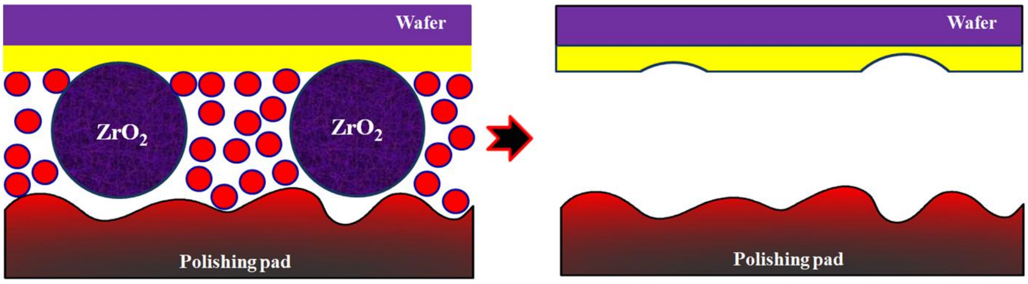


Fig. 16. Material removal capacity when polishing by CMS-2

4. CONCLUSIONS

Ionic and solid surface reaction modes in the CMS polishing process were investigated in this work. The experimental and analytical results show that the ionic surface reaction mode with the polishing process by CMS-2 gives better surface quality when polishing with the solid reactive from the polishing process by CMS-1 even though the two processes have the same reaction products. The main conclusions of the study are presented as follows:

The polishing results with CMS-2 show that the –Si–OH reactants are generated in the ionic form and are separated from the abrasive particles so that they can be adjusted to create a balance of chemical and mechanics action. The surface after polishing by CMS-2 was observed by SEM images and almost no scratches appeared on the surface.

The ionic surface reaction mode in the CMS-2 polishing process promotes the creation of a uniform reaction layer on the surface of the workpiece thereby promoting the ability to remove a uniform layer of material on the surface machining. The results show an ultra-smooth surface created by the polishing process with CMS-2.

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