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*chemical mechanical polishing,
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A NEW CHEMICAL MECHANICAL SLURRY FOR POLISHING YTTRIUM ALUMINIUM GARNET MATERIAL WITH MAGNESIUM OXIDE, SODIUM METASILICATE PENTAHYDRATE AND ZIRCONIUM DIOXIDE ABRASIVE PARTICLES

This work provided a new chemical-mechanical polishing mixture with MgO, sodium metasilicate pentahydrate, ZrO₂ abrasive particles, and deionized water. With chemical-mechanical slurry (CMS) proposed for polishing yttrium aluminum oxide (Y₃Al₅O₁₂) the surface reaction layer formed with significantly reduced hardness compared to other Y₃Al₅O₁₂ materials, these products combine with MgO to form montmorillonites (3MgO–Al₂O₃–3SiO₂–3Y₂O₃–5Al₂O₃). With this formation, the surface layer of Y₃Al₅O₁₂ material becomes soft and is easily removed by ZrO₂ abrasive particles under the influence of mechanical polishing, resulting in superfine surfaces generated from the proposed CMS model. The experimental results show that the surface quality with CMS proposed gives the surface quality with Ra = 0.471 nm along with the material removal rate 31 (nm/min). Surface quality is improved by 71% along with a superior material removal rate (increased by 287%) compared to silica slurry. The results show excellent polishing ability from CMS proposed for polishing Y₃Al₅O₁₂ materials.

1. INTRODUCTION

Nowadays, optical and laser devices are constantly evolving [1–3], and the demand for the fabrication of functional materials in this field is increasing, in which Y₃Al₅O₁₂ material has outstanding properties such as good optical properties, high thermal conductivity along with combinations of excellent rare earth ionic [4–6]. However, with difficult-to-machine properties such as high brittleness and hardness, it is difficult to achieve ultra-precise surface quality, with low surface quality that will reduce the efficiency of the laser, due to the low-quality surface will cause local energy accumulation for laser scattering [7, 8]. In addition, the cost of the Y₃Al₅O₁₂ material processing process also increases significantly due to its chemically stable nature thereby making the residual material removal rate low.

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In recent years various ultra-precision machining methods have been used to produce ultra-fine $Y_3Al_5O_{12}$ workpiece surfaces. The main methods used include chemical, magnetorheological finishing, mechanical, and chemical mechanical slurry (CMS) polishing [9, 10]. When mechanical and magnetorheological polishing for $Y_3Al_5O_{12}$ material, it is necessary to use abrasive particles with a higher hardness than $Y_3Al_5O_{12}$, in this case, scratches appear along with which the $Y_3Al_5O_{12}$ workpiece surfaces are easily displaced and appear large protrusions after polishing [11, 12]. Meanwhile, chemical polishing processes with $Y_3Al_5O_{12}$ material by phosphoric acid at high temperature and concentration will adversely affect the environment and equipment, in addition, create corrosion pits on the surface after polishing along with the ability to remove residuals on the surface of uneven workpieces [13]. These characteristics suggest that mechanical or chemical polishing alone is not suitable for polishing $Y_3Al_5O_{12}$ material. Studies by Mu et al. [12] show that when combined mechanical and chemical action in polishing processes can produce high surface quality by soft abrasive particles. In the process of polishing by CMS, the composition of the CMS has a great influence on the cost and surface quality [14, 15], the current studies mainly focus on polishing $Y_3Al_5O_{12}$ material by colloidal silica. $Y_3Al_5O_{12}$ material surfaces can be made in nanometer form when polished by chemical cloth and colloidal silica. The studies by Li et al. [16] showed that the surface of $Y_3Al_5O_{12}$ material was significantly improved when polishing by CMS containing a mixture of SiO_2 and NaOH suspensions, but the removal capacity of the workpiece residue was low at 0.29 (nm/min). Besides, another factor affecting the use of colloidal silica is that the ability to remove the processing residue is reduced in the process of reusing colloidal silica to produce $-Si-OH$ [17, 18]. Several studies have been conducted to improve the performance of colloidal silica, however, the performance improvement is still limited [19]. As such, there are still many problems to be solved in CMS polishing for $Y_3Al_5O_{12}$, which necessitates the creation of a new CMS blend for efficient material removal and improved surface quality better.

The polishing process uses a mixture of colloidal silica, mechanical polishing processes and chemical reactions are grouped together because the $-Si-OH$ produced are distributed on the surface of the silica particles as a reactant. With this feature, the polishing process performance is degraded because there is unbalance between chemical and mechanical polishing effects, therefore need to generate the Si-OH group in ionic form instead of solid form on abrasive particles. This process is solved by Na_2SiO_3 solution, in which Si-OH groups are generated by hydrolysis of SiO_3^{2-} [20], however, in these studies, the equilibrium condition between mechanical and chemical action must be determined, which is time-consuming and difficult to determine in polishing processes.

Aim to overcome the shortcomings in the chemical-mechanical polishing processes as analyzed above, this study develops a more effective chemical-mechanical polishing compound. Polishing mix is set based on the $-Si-OH$ components are separated from the abrasive particles created by the $Na_2SiO_3 \cdot 5H_2O$ solution along with the mechanical process performed by the ZrO_2 abrasive. More importantly, the proposed CMS polishing processes have added MgO to further enhance material removal as well as improve surface quality. Experimental processes have been performed showing that CMS containing percentage composition of $Na_2SiO_3 \cdot 5H_2O$, ZrO_2 , and MgO of 5%, 8%, and 1%, respectively, gives the highest polishing efficiency of $Y_3Al_5O_{12}$ material.

2. MATERIALS AND METHODS

The $Y_3Al_5O_{12}$ workpieces used in CMS polishing are diameter and high of 16 mm and 6 mm. The materials used for CMS polishing include deionized water, $Na_2SiO_3 \cdot 5H_2O$ with a purity of 99.9%, MgO and ZrO_2 with average particle size 50 nm. Commercial colloidal silica containing 10% by weight SiO_2 was used as a control in CMS. Experimental procedures were performed on a LAM-PLAN polishing machine as shown in Fig. 1. The machining parameters of the grinding and polishing processes are described in Table 1.

Table 1. Setting parameters for grinding and polishing of $Y_3Al_5O_{12}$ material

Number	Characteristic	Force (N)	CMS flow rate (ml/min)	Speed (rpm)	Pad
1	Grinding	25	12	50	673LAA10-3M
2	Polishing	25	8	90	IC1000

In order to find the most effective CMS polishing compound for $Y_3Al_5O_{12}$ material, different slurries were investigated with the ingredients listed in Table 2. In case it is necessary to increase the amount of $-OH$, the mixtures are added with an appropriate amount of NaOH to give the required pH. The workpieces grinding and polishing processes are performed on the LAM-PLAN polishing machine. The polishing processes with CMS-1 to CMS-5 for the $Y_3Al_5O_{12}$ workpieces are carried out for 120 min and repeated three times.

Table 2. Setting different CMS polishing mixes

CMS	pH	MgO (%)	ZrO_2 (%)	$Na_2SiO_3 \cdot 5H_2O$ (%)	Solvent
CMS-1	–	0	8	0	Alcohol
CMS-2	3	0	8	0	Deionized water
CMS-3	13	0	8	0	Deionized water
CMS-4	13	0	8	5	Deionized water
CMS-5	13	1	8	5	Deionized water

The surface morphology of $Y_3Al_5O_{12}$ material after polishing by different CMS mixtures for 120 min was observed by an MX-40 optical microscope. The surface quality and depth of scratches were measured on a Zygo NewView 7100 roughness test, the amount of material removed was determined by the scraping method.

Aim to investigate the polishing ability of $Y_3Al_5O_{12}$ workpiece by CMS-1 to CMS-5, the surface morphology was investigated at different locations in surface workpiece as depicted in Fig. 2. The obtained surface roughness is the average value at the four measurement locations.

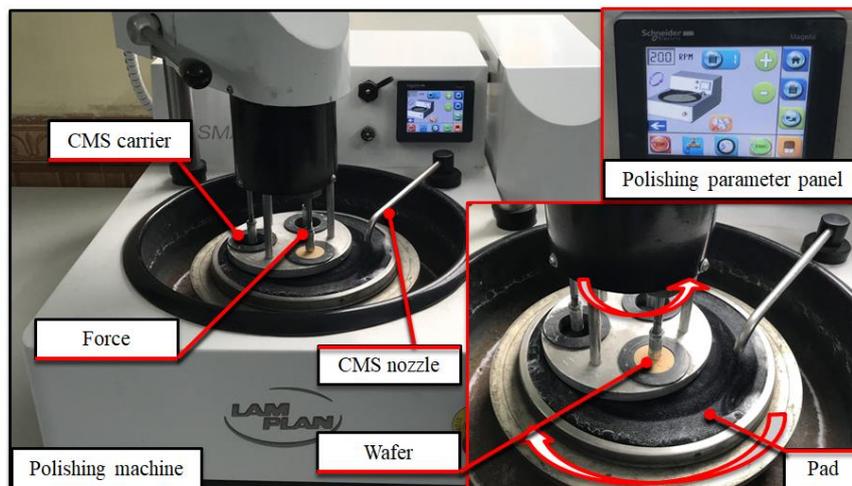


Fig. 1. Image experiment equipment by CMS polishing

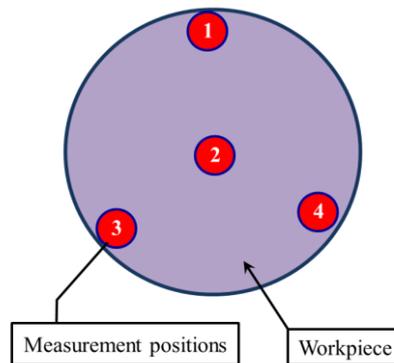


Fig. 2. Surface morphology analysis points

With CMS slurries used in the polishing process of $Y_3Al_5O_{12}$ materials as described in Table 2, the results show the ability to remove materials as described in Fig. 3. The results show that when the solvent used in CMS-1 is alcohol, the ability to remove the layer of material is negligible. It can be seen that the $Y_3Al_5O_{12}$ material has a higher hardness (with Moh 8.5) than the ZrO_2 abrasive particle (with Moh 7.5), so the mechanical polishing process is not effective. With the CMS-2 polishing compound, using deionized water as the solvent instead of alcohol showed a significantly increased ability to remove materials. In the solvent medium is deionized water, the reaction with $Y_3Al_5O_{12}$ creates a hydrated layer on the surface of the $Y_3Al_5O_{12}$ workpiece, ZrO_2 abrasive particles have a higher hardness than this layer, thus this layer is easily removed by abrasive particles. When adding the chemicals NaOH, $Na_2SiO_3 \cdot 5H_2O$, MgO to the deionized water, the material removal increased significantly. Thus, under the influence of these chemicals, beneficial reactions have been created for the material removed process. The surface morphology of $Y_3Al_5O_{12}$ material is shown in Fig. 4 with different CMS after 120 minutes of polishing, the results show that the surface quality and the ability to material removed are better improved when respectively adding components NaOH, $Na_2SiO_3 \cdot 5H_2O$, MgO into deionized water. Thus,

with the polishing method by CMS, the components of deionized water, NaOH, $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$, MgO have a very important role in the removal rate of materials and the surface quality of YAG materials.

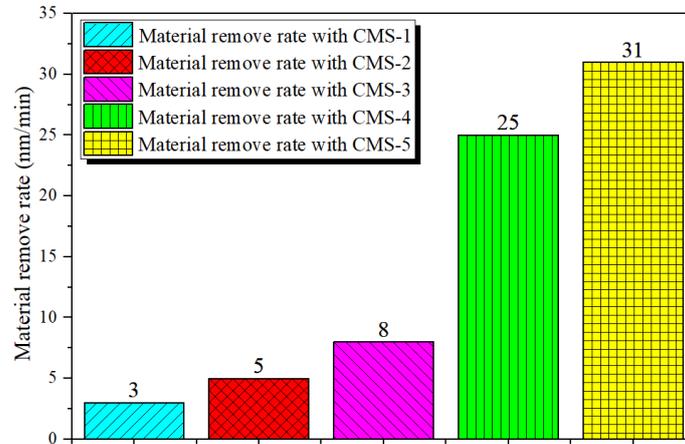


Fig. 3. Material removal capabilities with different CMS

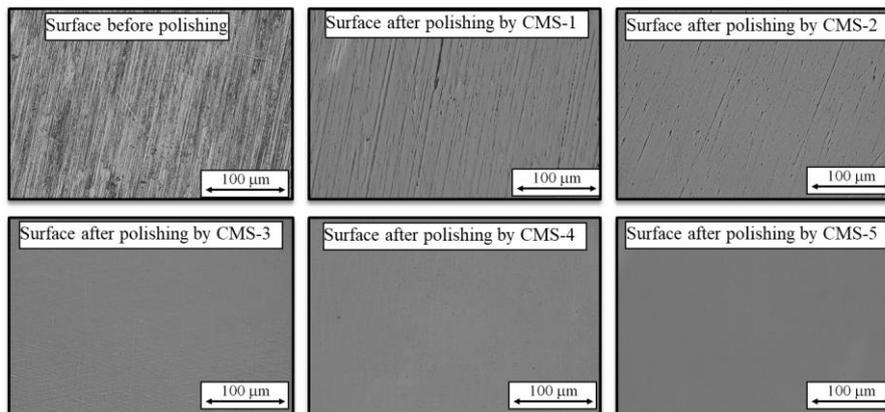


Fig. 4. Surface morphology of YAG material after polishing by different CMS

3. RESULTS AND DISCUSSION

3.1. POLISHED PERFORMANCE BY DIFFERENT CMS

The $\text{Y}_3\text{Al}_5\text{O}_{12}$ material was polished with the same treatment parameters by colloidal silica and the new CMS to investigate the performance of polishing slurries. The results of surface roughness and material removal are as described in Figs. 5 and 6. Surface quality results are obtained with $R_a = 1.637$ nm when polishing with silica while surface roughness with CMS is suggested for surface quality with $R_a = 0.471$ nm, thus quality surface has improved 71%. The material removal capacity when using colloidal silica is 8 (nm/min) while with the proposed CMS it is 31 (nm/min), thus with the proposed method for superior machining material removal (increase 287%) compared with silica slurry. The results show excellent polishing ability from CMS proposed for polishing $\text{Y}_3\text{Al}_5\text{O}_{12}$ materials.

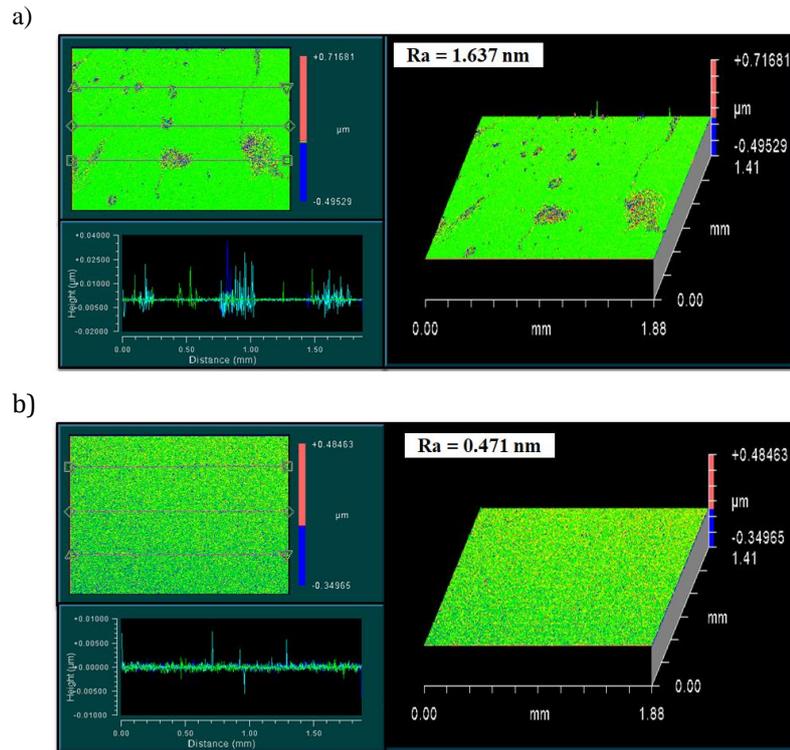


Fig. 5. Surface morphology of $Y_3Al_5O_{12}$ workpiece after polishing with different CMS: a) colloidal silica, b) new CMS polishing

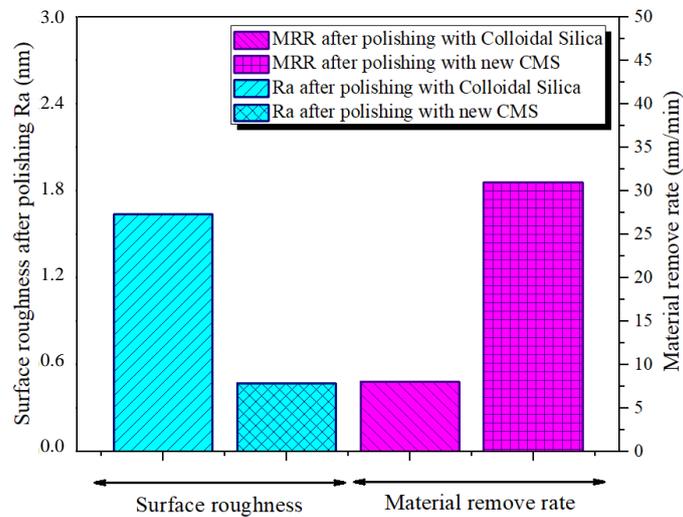


Fig. 6. Surface quality and material removal rate with different CMS slurries

3.2. HYDRATION REACTION

Many researchers have studied the interaction between the polishing mixture and the workpiece surface [21, 22]. In this study, the hydration reaction mechanisms on the workpiece surface were investigated. The generation of $AlOOH$ and $YOOH$ components is a result of the formation of $Al-OH$ and $Y-OH$ on the $Y_3Al_5O_{12}$ crystal surface. As depicted

in Fig. 7, due to the breakage of the Al–O and Y–O bond, a lot of voids are created for the O atom and unsaturated metal atoms are formed on the surface of the $Y_3Al_5O_{12}$ workpiece. Unsaturated metal atoms tend to adsorb H_2O by forming Al–O bonds and Y–O bonds. Meanwhile, H_2O dissociates into the H atom and –OH group. At this time, the O atoms will bond with neighboring H atoms on the $Y_3Al_5O_{12}$ surface and form the –OH group. Thus, two types of hydroxide groups in CMS and on $Y_3Al_5O_{12}$ surface bond together, forming AlOOH and YOOH. Besides, H_2O tends to dissociate into the –OH group in an alkaline environment, promoting hydration reaction [23, 24]. With this mechanism, CMS-3 has better polishing performance than CMS-2.

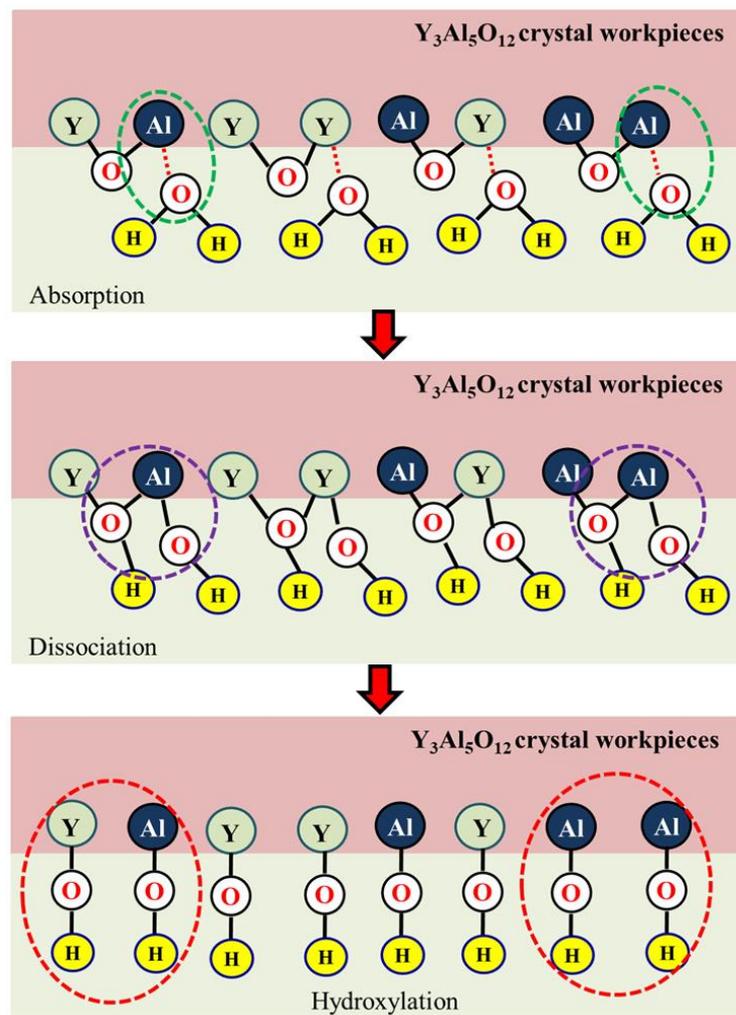


Fig. 7. Hydration reaction on $Y_3Al_5O_{12}$ workpiece surface

3.3. THE ROLE OF $Na_2SiO_3 \cdot 5H_2O$ IN THE POLISHING PROCESS WITH CMS

The XPS spectral descriptions as shown in Fig. 8 combine with the binding energy reference parameters listed in Table 3 [25, 26] show that different elements exist on the $Y_3Al_5O_{12}$ workpiece surface before and after polishing by CMS-4.

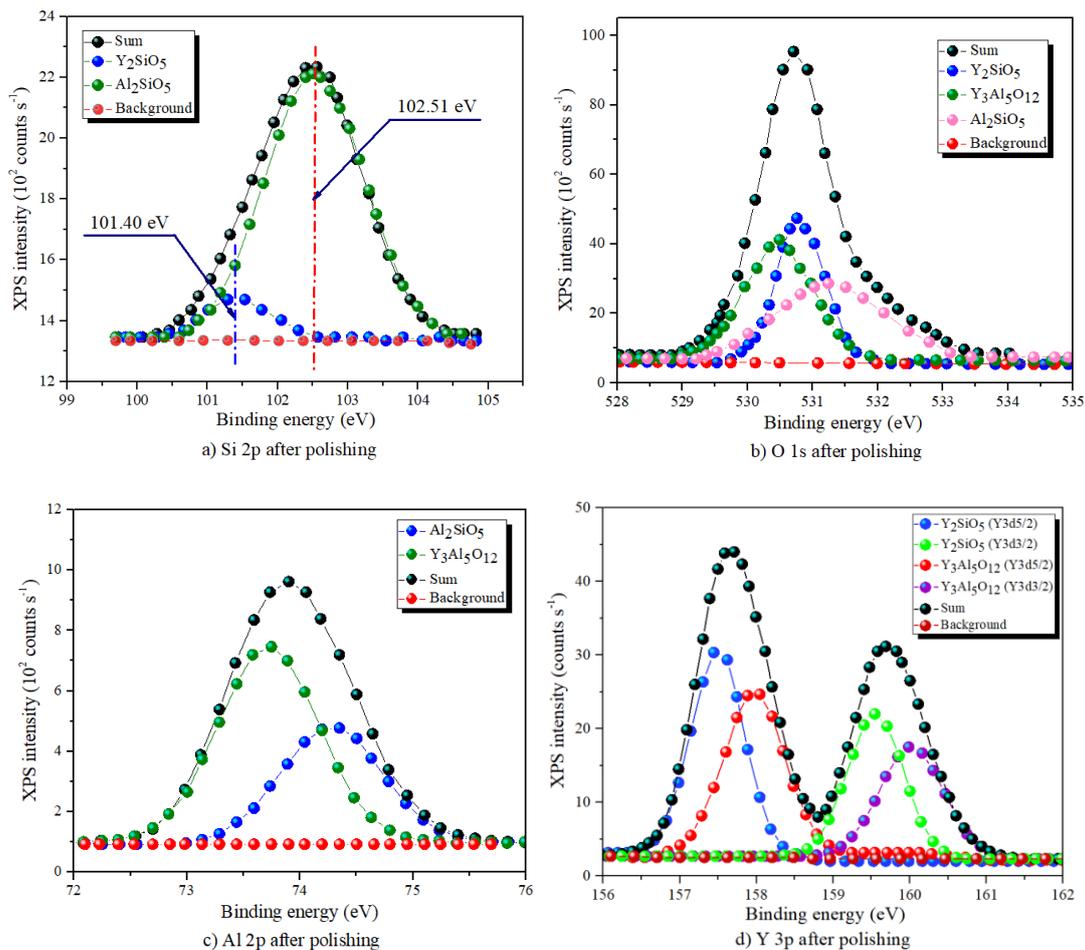
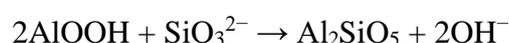
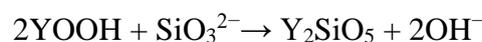


Fig. 8. Spectrum XPS after polishing

The analysis results of XPS spectra show that the changes of binding energies corresponding to Al_2SiO_5 and Y_2SiO_5 are generated on the surface of the $\text{Y}_3\text{Al}_5\text{O}_{12}$ workpiece during the polishing process. Figs. 8a and 8b depict the analysis of Si 2p and O 1s spectra after polishing of the surface layer with extreme points Si 2p (102.51 and 101.40 eV) and O 1s (531.3 and 53.80 eV) [27], respectively, this process shows that the products generated on the surface of the post-polished workpiece are Al_2SiO_5 and Y_2SiO_5 , respectively. Figs. 8c and d results of the peak analysis of Y 3d (158.01 and 160.05 eV) and Al 2p (74.27 eV) [28, 29] also correspond to the formation of Al_2SiO_5 and Y_2SiO_5 . With the presence of these materials on the $\text{Y}_3\text{Al}_5\text{O}_{12}$ workpiece surface layer, the material removal process is easier because the ZrO_2 abrasive particles have a higher hardness than the hardness of Al_2SiO_5 and Y_2SiO_5 . The dehydration mechanisms are as depicted in Fig. 9, in which the $\text{Y}_3\text{Al}_5\text{O}_{12}$ workpiece surface will react with the Si–OH group created by the SiO_3^{2-} reduction reaction. The Y–OH and Al–OH groups present on the surface layer of the workpiece tend to combine with the Si–OH group to form Y–O–Si and Al–O–Si bonds in deionized water. The hydration reaction mechanisms are described as follows:



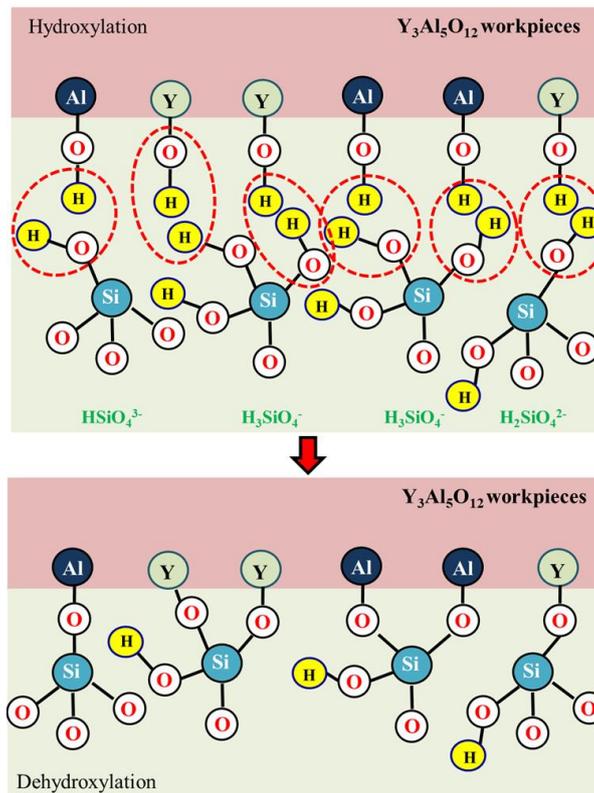


Fig. 9. Diagram of dehydration reaction

Table 3. Elemental binding energies

Chemical composition	Al 2p (eV)	Mg 1s (eV)	O 1s (eV)	Si 2p (eV)	Y 3d (eV)
$Y_3Al_5O_{12}$	–	–	530.47	–	157.43
Al_2SiO_5	74.27	–	531.30	102.51	–
Y_2SiO_5	–	–	530.80	101.40	158.00 160.05
$3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$	–	1305.30	–	102.95	–
MgO	–	1303.90	–	–	–

3.4. THE ROLE OF MgO IN THE PROPOSED CMS POLISHING PROCESS

After polishing by CMS-5, the results of XPS spectrum analysis with Mg 1s and Si 2p for the $Y_3Al_5O_{12}$ workpiece are as described in Fig. 10. The result in Fig. 10a obtained the peak value of Mg 1s (1305.3 eV) [30] which corresponds to the montmorillonite ($3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$) formed on the surface of the $Y_3Al_5O_{12}$ workpiece. Fig. 10b depicts XPS with Si 2p showing peaks at positions 102.51 eV, 101.40 eV, and 102.95 eV, the results show that the formation of Al_2SiO_5 , Y_2SiO_5 , and $3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$, respectively.

The results of the above analyses show that the production of the montmorillonite product on the surface layer results from the reaction of $Y_3Al_5O_{12}$ workpiece, Na_2SiO_3 , and MgO ingots in deionized water. Previous studies have shown that the participation of MgO will accelerate the formation of silicate minerals into montmorillonite ($3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$) trilayers [31, 32]. In which the secondary reaction between Al_2SiO_5 and MgO will form montmorillonite ($3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$) [33]. The resulting montmorillonite product is softer than Al_2SiO_5 and is therefore easily removed in mechanical polishing processes. This conversion benefits the CMS polishing process with improved surface quality as well as the ability to remove material residues.

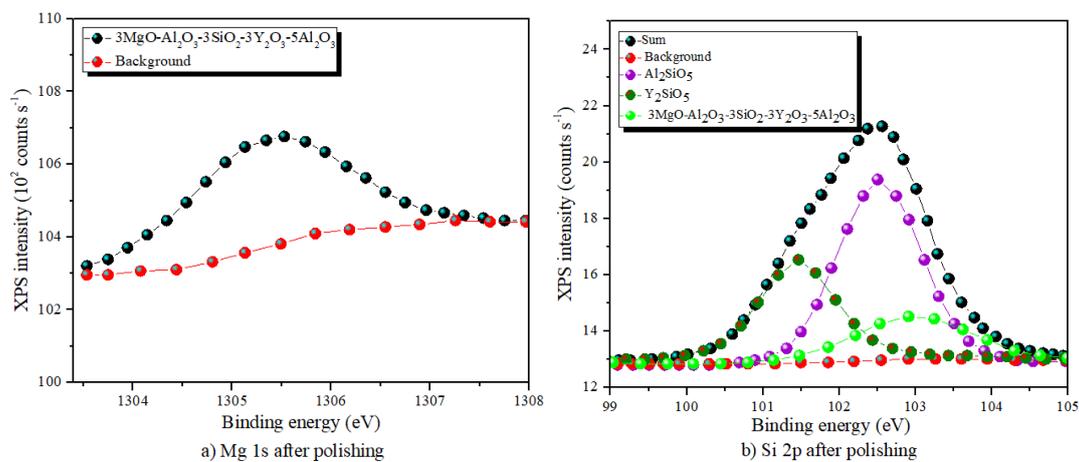


Fig. 10. Si 2p and Mg 1s XPS spectra after polishing $Y_3Al_5O_{12}$ workpiece by CMS-5

Experiments established by the proposed polishing model with CMS-5 have the ability to create ultra-smooth surfaces with high material removal rate for $Y_3Al_5O_{12}$ workpieces, thereby opening up many promises in material machining in the optic field. However, the current polishing model is only applied in polishing conditions with fixed technological parameters, along with that the workpiece surface is flat, and not applied to lens faces with complex profiles. In subsequent studies, the authors study the effect of the proposed CMS-5 mixture based on the reactions of the Si-OH group for other difficult-to-machine materials, along with the workpiece profiles complex. Processes for creating ultra-fine surfaces from $Y_3Al_5O_{12}$ workpieces and other lens materials are investigated with technological parameters optimized to reduce machining time and costs.

4. CONCLUSIONS

Aim to create the ultra-smooth $Y_3Al_5O_{12}$ workpiece surface, a new CMS polishing method was established with a mass percent composition including ZrO_2 (8%), $Na_2SiO_3-5H_2O$ (5%), MgO (1%), and deionized water. The results obtained after polishing by the proposed CMS-5 give out an ultra-smooth surface with high material removal capacity. The main conclusions being made as follows:

– The reaction mechanism was elucidated by X-ray photoelectron spectroscopy (XPS) analysis before and after polishing by different CMSs. Analytical results show that $Y_3Al_5O_{12}$ material produces YOOH and AlOOH in Na_2SiO_3 solution, then combines with –Si–OH to form (Y-Si) and (Al-Si) with significantly reduced hardness compared to other $Y_3Al_5O_{12}$ materials, these products combine with MgO in CMS-5 proposed to form montmorillonites ($3MgO-Al_2O_3-3SiO_2-3Y_2O_3-5Al_2O_3$). With this formation, the surface layer of $Y_3Al_5O_{12}$ material becomes soft and is easily removed by ZrO_2 abrasive particles under the influence of mechanical polishing, resulting in superfine surfaces are generated from the proposed CMS-5 model.

– The newly developed CMS-5 polishing compound has produced an ultra-smooth surface with $R_a = 0.471$ nm along with a high workpiece residue removal of 31 nm/min. With the CMS-5 proposed method, the surface quality has improved by 71%, and superior machining material removal (an increase of 287%) compared with silica slurry. The results show excellent polishing ability from CMS proposed for polishing $Y_3Al_5O_{12}$ materials. The model polishing with CMS-5 has been successfully experimented exhibiting feasibility and high applicability.

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The data used to support the findings of this study are included in: <https://assets.researchsquare.com/files/rs-1198259/v1/035e3351-e9d1-43cf-9d03-9b3f3f7567fc.pdf?c=1653395797>

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