Machining of Single-Crystal Sapphire with Polysaccharide-Bonded Abrasive Tool

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Abstract: A novel polysaccharide-bonded abrasive tool is proposed for the green machining of single-crystal sapphires. The prescription and manufacturing process of the proposed tool is designed, and the gelation property of polysaccharide by microwave treatment is investigated. Abrasive tool samples are fabricated, and a machining experiment on a single-crystal sapphire is performed. It is found that the crystallinity of polysaccharide gel decreases as the proportion of cross-linked polysaccharide increases. Abrasive tool samples with cross-linked polysaccharide present higher surface hardness. With the new abrasive tool, the surface quality of sapphire wafer can be significantly improved. This new tool with an abrasive to binder ratio of 2∶1 attains a material removal rate of 0.68 μm/min. It is found that increasing the abrasive to binder ratio leads to better self-dressing performance but worse material removal ability and greater loss of abrasive tool materials. The validity of polysaccharide as an abrasive tool binder is preliminarily verified.

Key words: polysaccharide binder; microwave treatment; single-crystal sapphire; green manufacturing

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0 Introduction

Surface machining, such as the back-grinding and polishing of a single-crystal sapphire substrate, is an important step in the manufacturing process of advanced lighting chips. It requires high machining efficiency and surface quality[1-2]. In response to the rapidly increasing market demand, the productivity in the precision machining of wafer substrates has remarkably increased in recent years; however, it has also introduced negative environmental effects that cannot be ignored[3]. Currently, the realization of high-quality and high-efficiency green machining of semiconductor substrate wafers is one of the key problems in ultra-precision machining[4-5].

An effective technique for the ultra-precision machining of single-crystal sapphire wafer is chemo-mechanical grinding (CMG)[6-7]. With this new method, the surface roughness of a machined sapphire surface can have a R_a value of less than 1 nm, and the material removal rate can reach 0.22 μm/min[7]. By employing the mechano-chemical effect, the material removal with high surface integrity can be achieved[8-9]. Currently, the abrasive tool binder in the CMG is typically phenolic resin whose raw materials are toxic oil derivatives. In view of this, the use of phenolic resin in the manufacture of abrasive tools and machining endangers human health and environment[10]. Moreover, because oil is non-renewable, the long-term use of phenolic resin is unreliable. In recent studies, efforts have been made to investigate new types of binders, such as sodium alginate hydrogel[11] and magnesium oxychloride[12], for abrasive tools to improve

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their environmental friendliness and machining performance.

Polysaccharide, with hydroxyl functional groups in its molecular structure, is a type of high polymer that can be prepared from natural products. Starch is the most common natural polysaccharide, and various modification processes (cross-linked, oxidation, etc.) can significantly improve its strength and stability. Modified polysaccharide has been widely used in products, such as environment-protecting tableware, adhesives, and casting, as well as in 3D printing materials[13].

In this paper, a novel polysaccharide-bonded abrasive tool is proposed. The prescription and manufacturing process of this proposed abrasive tool is designed, and the gelation properties of polysaccharide by microwave treatment are also investigated. A sample of the abrasive tool is manufactured, and a machining experiment on a single-crystal sapphire using the new abrasive tool is performed. The surface textures of abrasive tool samples are observed, and the validity of polysaccharide binder is discussed.

1 Polysaccharide

1.1 Natural polysaccharide

In the natural world, natural polysaccharide (NP) widely exists in plants, animals, and microorganisms. The molecular structure of the adopted polysaccharide is a single-glucose chain connected by glycosidic linkages, as shown in Fig.1. There are three alcoholic hydroxyls in a single glucose unit: C6 primary alcoholic hydroxyl, and C2 and C3 secondary alcoholic hydroxyls. Alcoholic hydroxyl has good reactivity and can create hydrogen bonds, which can link neighbouring polysaccharide units and provide the polysaccharide with high cohesive strength.

1.2 Cross-linked polysaccharide

To further improve the abrasive tool strength, cross-linked polysaccharide (CLP) is adopted and mixed with native polysaccharide, which functions as the binder. Cross-linking is a common method

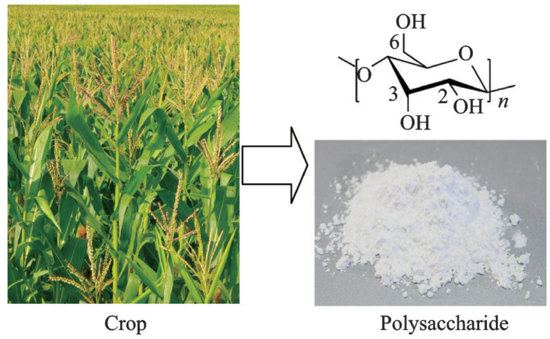

Fig.1 Polysaccharide from natural product

used in the preparation of gel materials[14]. By cross-link modification, the alcohol hydroxyls of polysaccharide can react with reagents having dual or multiple functional elements. Hydroxyl groups from different polysaccharides can therefore connect with each other to form a reticulated structure, which improves the abrasive tool's mechanical strength. The most familiar cross-link agent is epichlorohydrin or sodium trimetaphosphate. For example, when epichlorohydrin is employed as the cross-linking agent, the cross-link reactions shown in Fig.2 will occur at pH 10 and 30 ℃.

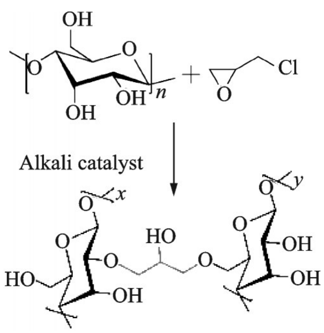

Fig.2 Cross-linking of polysaccharide by epichlorohydrin[15]

1.3 Gelation of polysaccharide by microwave treatment

To use polysaccharide as binder, it has to be heated in aqueous environment until the micro-particles that swell with its crystalline area disappear (this process is called gelation). The glucose chains thereafter disperse in water, attach on the abrasive surface, and link with each other through the hydrogen bond to form a gel network. When the aqueous system's temperature decreases, free glucose chains regularly cluster through hydrogen bonds and form microcrystallines. This process is known as recrystallisation.

In this study, the gelation by microwave treatment is adopted. Compared with traditional polysaccharide gelation methods, such as the hydrothermal gelation, the microwave treatment is time-saving and results in even gelation. The gelation and recrystallisation of polysaccharide are crucial to achieve the mechanical properties of polysaccharide-bonded abrasive tools. A gelation experiment is accordingly performed with the prescribed design listed in Table 1.

Table 1 Designed prescriptions of gelation experiment

No.	NP /g	CLP /g	Water /g
A	15	0	30
B	7.5	7.5	30
C	3	12	30

For each prescription listed in Table 1, four different microwave treatment parameters are adopted: #1(P100T20), #2(P80T40), #3(P40T80), and #4(P20T100). Here, the microwave oven output power is 900 W, *P* represents the percentage of the full output power, and *T* is the heating time (s).

The gelation in the experiment is achieved as follows. Polysaccharide is added to the distilled water. Heating and mixture gelation are realized by microwave treatment with a working frequency of 2 450 MHz. After heating, the single glucose chains link with each other and form a highly viscous gel. The gel is thereafter exposed to air (ambient temperature is 10 ℃) for 24 h and then placed in an incubator with a temperature of 3 ℃ for 4 h. Finally, the gel is dried by hot air until its weight becomes constant. The gel dries into a hard semi-transparent solid film.

It is found that gel samples with cross-linked polysaccharides have a higher hardening speed, and the cross-linked polysaccharide improves the strength of polysaccharide gel. On the other hand, the gel crystallinity also affects the strength of the polysaccharide binder. The gel samples are accordingly tested by X-ray diffraction (XRD) (DX-2700, Haoyuan Instrument Co., China; step size: 0.02; scan speed: 1°/min). The XRD results of the gel with prescription *B* and the dispersion peak caused by the microcrystalline of polysaccharide are shown in Fig.3. There is a strong peak near the diffraction angle of 17° and two medium peaks near 22° and 24°, indicating that the crystal is *B*-type form[16].

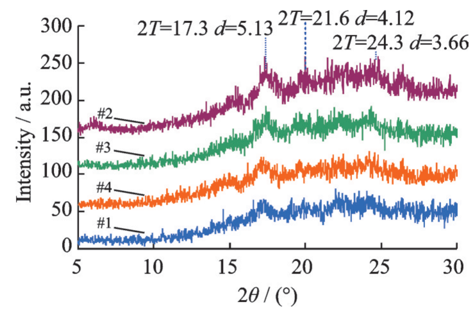

Fig.3 XRD spectrum of gel with prescription *B*

It can be observed in Fig.3 that the gel samples mainly have an amorphous structure with an extremely low crystallinity. Their crystallinity can be calculated by

$$X_C = \frac{I_C}{I_C + I_A} \tag{1}$$

where X_C, I_C, and I_A are the crystallinity, cumulative diffraction intensity of crystalline, and amorphous area, respectively[17]. The calculation results are shown in Fig.4.

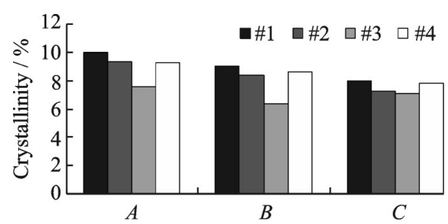

Fig.4 Gel crystallinity with different prescriptions

Fig.4 shows that the gel crystallinity decreases as the proportion of cross-linked polysaccharide increases. This occurs because the cross-linked polysaccharide can delay the polysaccharide recrystallisation[18]. In the following composition design of polysaccharide-bonded abrasive tool, the ratio of the natural polysaccharide to cross-linked polysaccharide is accordingly set to be 1∶1. It is also found that samples with P100T20 microwave treatment have the highest crystallinity.

2 Polysaccharide-Bonded Soft Abrasive Tool

2.1 Composition

The composition details of the new abrasive tool are summarised in Table 2. The abrasive to binder ratios (ABRs) are 2∶1, 4∶1, and 6∶1, and the natural/cross-linked polysaccharide proportions are 1∶0 and 1∶1. For each prescription listed in Table 2, three different microwave treatment parameters are adopted: #1(P100T20), #2(P80T40), and #3(P20T100).

Table 2 Prescription of polysaccharide-bonded abrasive tool

No.	NP /g	CLP /g	Abrasive /g	Water /g
a	2	0	4	5
b	2	0	8	7
c	2	0	12	8
A	1	1	4	4
B	1	1	8	6
C	1	1	12	8

The selected abrasives are #600 SiO_2 spherical beads with a hardness of 2 on the Mohs' scale (the so-called soft abrasive). Under a certain contact pressure and relative speed, the mechano-chemical reaction between SiO_2 and sapphire occurs as follows[19].

$$3\alpha - Al_2O_3 + 6SiO_2 = 3\alpha - Al_2O_3 \cdot 2SiO_2 \quad (2)$$

$$\alpha - Al_2O_3 + SiO_2 = \alpha - Al_2O_3 \cdot SiO_2 \quad (3)$$

The resultants of the above reactions are mullite or cyanite with a hardness of 6—7 on the Mohs' scale that can be removed by SiO_2 abrasives. Thus, the non-damage material removal of sapphire can be realised.

2.2 Manufacturing process

The manufacturing process of polysaccharide-bonded soft abrasive tool is shown in Fig.5.

After full stirring, the distilled water is added to the mixed powder of polysaccharide and abrasive. The mixture has to be heated until the polysaccharide swells and becomes single molecules because polysaccharide does not dissolve in water. The heating and gelation are realized by microwave treatment with a working frequency of 2 450 MHz. After heating, the single glucose chains link with each other and form a highly viscous gel. A refrigeration step of 3 ℃ with a polypropylene (PP) film cover is required. After the refrigeration, the gel is subjected to drying using alcohol displacement and hot air until the weight of the gel becomes constant. As shown in Fig. 5, all the raw materials used during the manufacturing process are non-toxic, and all the solvents are waterborne. Compared with the manufacturing process of the abrasive tool using resin binders, such as phenolic resin, the manufacturing process of the new abrasive tool is environment-friendly.

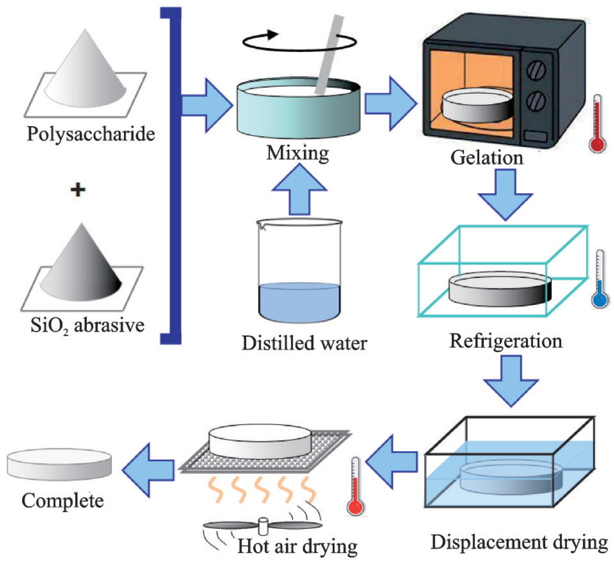

Fig.5 Manufacturing process of polysaccharide-bonded abrasive tool

The abrasive tool samples with no cross-linked polysaccharides are more severely warped than those with cross-linked ones. The surface hardness of the abrasive tool samples is measured by a LX-D digital shore durometer and the number of sampling points is four. The measurement results (except for sample #*c*-3 because of its poor forming quality in the boundary area) are presented in Fig.6.

Fig. 6 shows that the surface hardness of abrasive tool samples decreases with the increase in abrasive-binder ratio, and the samples with cross-linked polysaccharides have a higher surface hardness. This indicates that cross-linked polysaccharides improve the abrasive tool's strength.

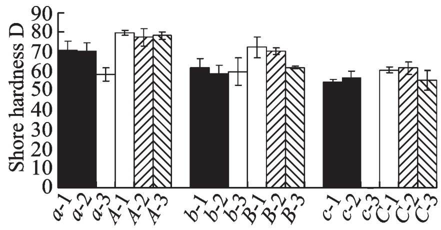

Fig.6 Surface hardness of abrasive tool

3 Machining Experiment of Sapphire Wafer

3.1 Machining experiment setup

To study the machining performance of the new abrasive tool, a machining experiment is performed on a single-crystal sapphire wafer using a 10-mm diameter and 0.7 mm thick multi-wire saw. The crystal plane for machining is C plane (0001). The experimental setup is shown in Fig.7. The sapphire wafer is fixed on an auto-maintain-load platform. The machining parameters are summarized in Table 3. If the sapphire wafer is parallel to the face of abrasive tool, the surface layer of the abrasive tool may be scraped by the edge of the wafer during

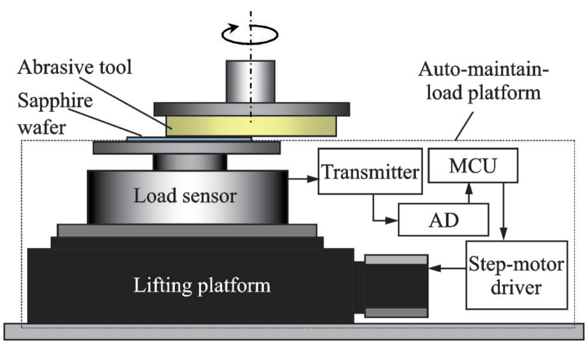

Fig.7 Experiment setup

Table 3 Machining parameters

Parameter	Value
Rotating speed of abrasive tool /($r \cdot min^{-1}$)	566
Load /N	20
Cooling	Dry machining
Machining time/min	30

friction. This can affect the evaluation of the abrasive tool's self-sharpening performance. The sapphire wafer is accordingly set to be slightly tilted towards the abrasive tool with a sectional shape friction area. Abrasive tool samples with no cross-linked polysaccharides are not utilized in the machining experiment because of their poor forming quality.

3.2 Experimental results

After the experiment, the machined surface of sapphire wafer is cleaned with ethanol and distilled water. The machined areas of sapphire wafers are observed by optical microscopy (XZJ-2030, Phoenix, China), as shown in Fig.8. In the boundary area of *A*-2, the rough surface marks created by wire saw cutting can be observed. The machined area exhibits a considerably smoother surface. Each of the nine samples of the abrasive tool can significantly improve the surface quality of sapphire wafer. Grooves with no brittle cracks are formed by the scratching of SiO_2 abrasives because the sapphire wafer is fixed as the abrasive tool rotates. During this period, the sapphire material is removed by the mechano-chemical reaction between SiO_2 abrasives and sapphire. The *C*-series samples present the best machining quality among the samples with considerably few grooves.

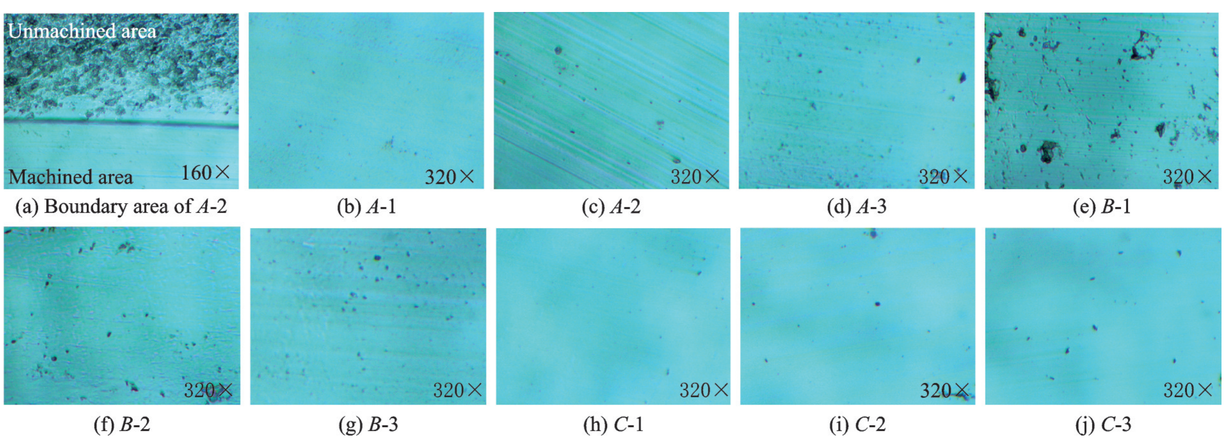

Fig.8 Surface texture and roughness of machined sapphire wafer

The profile of the abrasion areas on the sapphire wafer surface is measured by a profilometer (SV3200, Mitutoyo, Japan). A typical profile of a machining spot is shown in Fig.9, where the removal depth of sapphire materials can be determined. The removal depth of different abrasive tools is shown in Fig.10. This indicates that this depth generally decreases as the ABR increases. Among the *A* and *B* samples, those that have been subjected to P80T40 microwave treatment present a lower material removal depth, which may be related to the crystallisation of polysaccharide binder. The maximum removal depth with a value of 20.51 μm is created by *A*-1 abrasive tool. The material removal rate of *A*-1 abrasive tool attains 0.68 μm/min over a 30 min machining time. The machining results indicate that the application of polysaccharide as an abrasive tool binder is feasible.

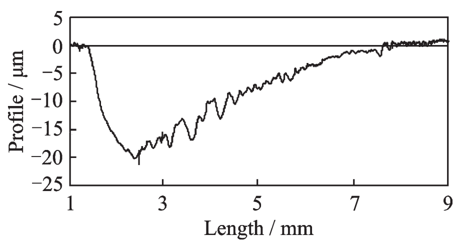

Fig.9 Section profile of machining spot by abrasive tool *A*-1

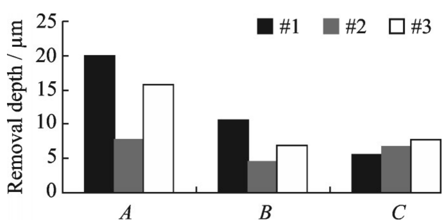

Fig.10 Removal depth by different abrasive tools

4 Discussion

Abrasive tool samples after machining are shown in Fig. 11. An annular yellowish scorching mark appears in the abrasion area of *A*-series samples. Scattered scorching marks are also distributed on the surfaces of *B*-series samples. Scorching marks are not found on *C*-series samples.

The surface textures of the abrasive tools are observed by XZJ-2030 microscopy with a magnification of 160× in polarisation mode. The direct observation of SiO_2 grits and consolidated polysaccharides is difficult because they are all transparent. Here, iodine tincture is utilized because triiodide ion can cause complexities in polysaccharide glucose chains and modify the light-absorbing property of polysaccharide[20]. When the iodine tincture is smeared, the polysaccharide binder becomes purple, and the bonded spherical SiO_2 abrasives can be distinguished by cross-extinction under polarised light. For the series of *A* and *B* samples, iodine tincture is smeared on the scorching areas. The observation results of samples *A*-1, *B*-1, and *C*-1 are shown in Fig.12.

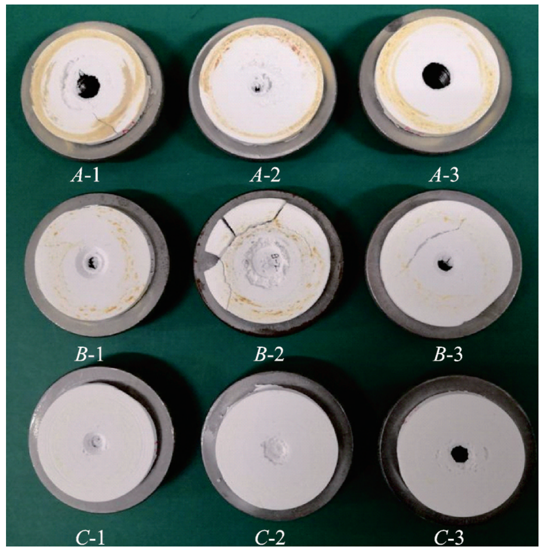

Fig.11 Abrasive tool after machining

The cross-extinction of spherical SiO_2 abrasives is indicated by yellow arrows in Fig. 12. The figure shows that before the machining experiment, the distribution density of SiO_2 abrasive increases with the ABR. After machining, a blocking area with few abrasives is observed on the surface of sample *A*-1. Compared with the initial surface, it is observed that sample *B*-1 has few blocking areas with a lower distribution density of abrasives. There is practically no change observed on the surface texture of sample *C*-1. It seems that the *C*-series samples have the best self-dressing performance.

The powder produced during the machining process is thereafter collected, as shown in Fig.13. From *A* to *C*, the amount of powder increases, indi-

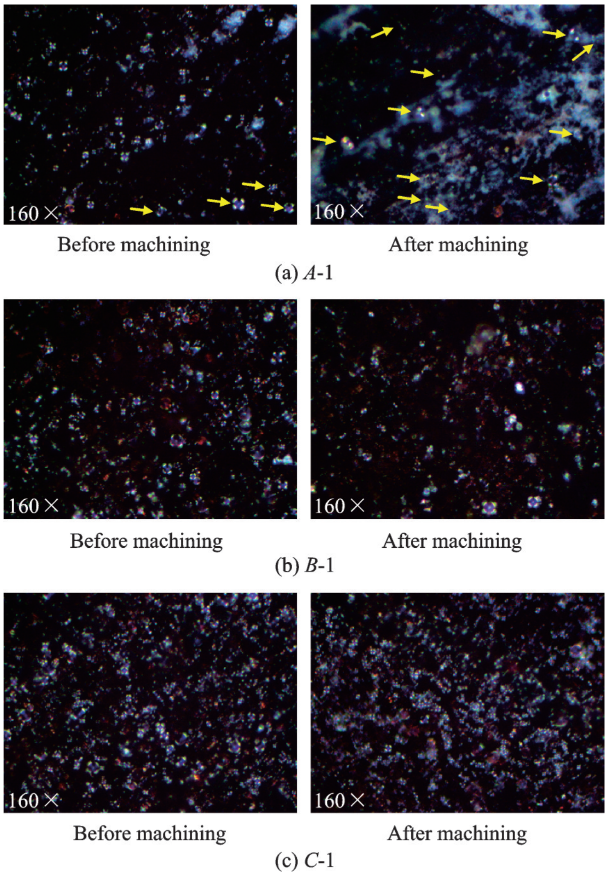

Fig.12 Micro-textures of abrasive tool surface

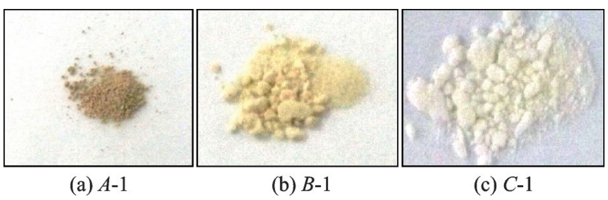

Fig.13 Powder produced in machining process

cating that abrasive tool materials are lost. As the ABR increases, the abrasive holding ability of the polysaccharide binder becomes weaker. This means that the abrasive easily falls off from the tool surface during the machining process. This explains why the *C*-series samples have the best self-dressing performance and lowest material removal ability.

From *A* to *C*, the powder colour also becomes lighter. During the machining process, the friction heat is produced in the frictional region between the polysaccharide binder and the sapphire. The polysaccharide partly converts to furaldehyde, especially 5-hydroxymethylfurfural, by dehydration under high temperatures[21-22]. The furaldehyde thereafter becomes yellow and further transforms rapidly into a brown resin-like polymer under the action of the heat and oxidation. In Fig.13, the powder colour of samples from *A*-1 to *C*-1 becomes lighter (from brown to greyish white). The reason lies in that the *A*-series samples have the best abrasive holding ability, a higher temperature can be reached during the machining, and a larger amount of furaldehyde is generated in the powder.

The powders produced during the machining process with different abrasive tools are observed by field emission scanning electron microscopy (SEM) (FEI Inspect F50, USA), whose results are shown in Fig.14.

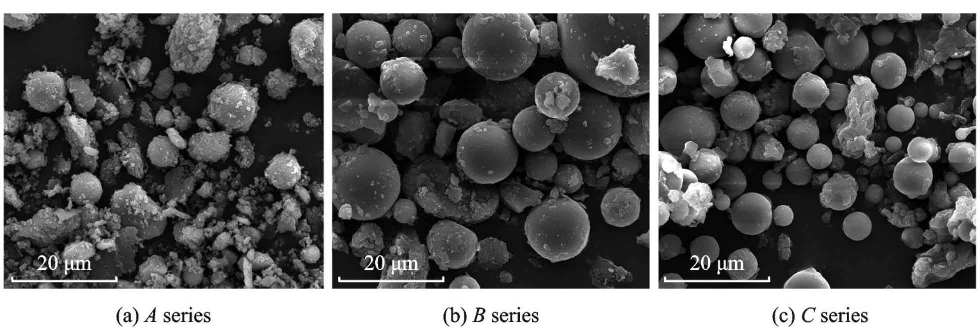

Fig.14 SEM results of powder produced during machining process

It can be indicated from Fig.14 that as the ABR increases, there are more dropped abrasives appearing in the powder, and the surface of dropped abrasive becomes smoother. It means that the depth of the polysaccharide binder attached to the SiO_2 abrasive surface decreases, and the sectional area of the bonding link between adjacent abrasives also decreases as the ABR increases. As a result, the bonding strength between adjacent abrasives and the ability of the tool to hold the abrasives decrease, thus affecting the machining and self-dressing performance of abrasive tools.

The SEM image of single abrasive in the powder produced by the *A*-series abrasive tool is shown in Fig.15. It can be observed that the polysaccharide binders are evenly attached to the abrasive surface, indicating that polysaccharides have good compatibility with SiO_2 abrasives. It can also be concluded that the abrasives fall off because of cohesive fractures between adjacent polysaccharide binders and not the result of adhesives fractures between polysaccharide binders and SiO_2 abrasives. The low bonding strength of the polysaccharide limits the ability of the tool to hold the abrasives.

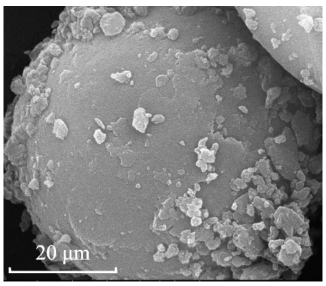

Fig.15 SEM image of single abrasive in powder produced by *A*-series abrasive tool

To further study the influence of ABR on the machining performance of abrasive tools, the powders produced during the machining process employing different abrasive tools are analysed by energy dispersive spectroscopy (EDS) (QUANTAX 100, BRUKER, USA). The EDS results are shown in Fig.16.

In Fig.16, the two 2 keV peaks are caused by the Au element introduced through the gold-spraying process. The Si and O elements mainly belong to the SiO_2 abrasives that fell off. The Al element is a solid-solid reaction product between SiO_2 abrasives and single-crystal sapphire. The carbon element mainly belongs to the polysaccharide binder attached to the abrasives that fell off. As the ABR increases, the peak intensity of the Al element decreases, proving that the increase in the ABR decreases the abrasive tool's holding ability. This reduces not only the heat generated in the friction area between the abrasive and single-crystal sapphire but also the reaction rate of the solid-solid reaction product between SiO_2 abrasives and single-crystal sapphire. The peak intensity of carbon element also decreases with the ABR increase. This similarly indicates that the higher the ABR, the lower the bonding strength among abrasives, and the lower the abrasive holding ability of tools.

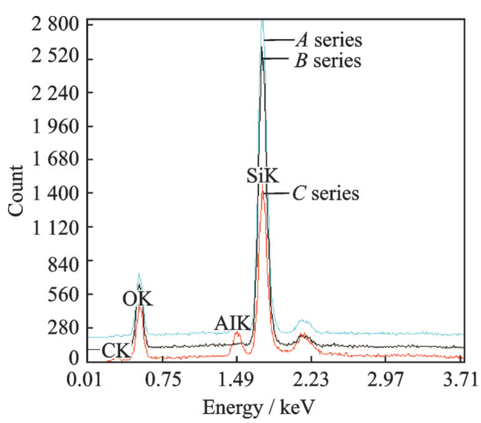

Fig.16 EDS results of powder produced during machining process

By considering the micro-texture of a single-crystal sapphire machined by different abrasive tools, it can be deduced that for abrasive tools with different ABRs, the material removal mechanics differ. The *A*-series abrasive tools with an ABR of 2∶1 have better abrasive holding ability, and the materials are mainly removed by the grooving process (twin body wear). The *C*-series abrasive tools with an ABR of 1∶6 have worse abrasive holding ability, and the materials are mainly removed by the rolling and micro-grooving of abrasives that fell off (three-body wear). This explains why the *C*-series samples present the best quality in the machining of sapphire wafer among the abrasive tool samples.

5 Conclusions

A novel polysaccharide-bonded abrasive tool for green surface machining of single-crystal sapphire is proposed. The following conclusions can be drawn.

(1) The crystallinity of polysaccharide gel decreases as the proportion of cross-linked polysaccharides increases. The prescription of the polysaccharide-bonded abrasive tool with SiO_2 abrasives is designed, and the manufacturing process of the new abrasive tool is established. The composition and manufacturing process of the new abrasive tool are

fully environment-friendly. Abrasive tool samples with various ABRs are made. The surface hardness of abrasive tool samples decreases with the increase in the ABR, and the samples with cross-linked polysaccharides present a higher surface hardness.

(2) The machining of single-crystal sapphire wafer based on the new abrasive tool is performed, and results show that all the abrasive tool samples can significantly improve the surface quality of sapphire wafer. The removal depth generally decreases as the ABR increases. The material removal of abrasive tools with an ABR of 2∶1 attains the rate 0.68 μm/min. The machining results indicate that the application of polysaccharide as an abrasive tool binder is feasible.

(3) As the ABR increases, the abrasive holding ability of polysaccharide binder becomes weaker, leading to a better self-dressing performance of the abrasive tool. Moreover, the material removal ability decreases, and the material loss of the abrasive tool increases.

In the future study, the balance between the material removal and self-dressing abilities of the polysaccharide-bonded abrasive tool should be investigated. Furthermore, the prescription of the new abrasive tool has to be modified to improve its mechanical strength.

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采用多糖结合剂磨具的单晶蓝宝石加工

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摘要:提出了一种用于单晶蓝宝石绿色加工的新型多糖结合剂磨具。设计了磨具的配方和制备工艺,研究了微波处理对多糖凝胶性能的影响。制作了磨具试样并对单晶蓝宝石进行了加工试验。研究发现,随着交联多糖比例的增加,多糖凝胶的结晶度降低。含有交联多糖的磨具试样具有较高的表面硬度。加工试验结果表明,采用这种新型磨具,蓝宝石晶片的表面质量得到了显著的改善。当磨具砂结比为2:1时,材料去除率可达0.68 μm/min,提高磨具砂结比可获得较好的自锐性能,但材料去除能力降低,磨具磨耗增大。初步验证了多糖材料作为磨具结合剂的有效性。

关键词:多糖结合剂;微波处理;单晶蓝宝石;绿色加工