DEVELOPMENT OF A COMPOSITE GRINDING STONE FOR AN USE OF ULTRA PRECISION PROCESSING OF GLASS

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SUMMARY : A glass plate of ultra fine mirror finished surface is processed by a newly developed grinding stone in which ultra fine particles of cerium oxide(CeO_2) are fixed by a special fixing method. Fine particles of CeO_2 are previously immobilized in micro spherical cells, then are fixed into matrix of grinding stone. The mirror finished surface can be obtained by the developed grinding stone, and an insufficient removal rate can be recovered by intermittent supply of a small amount of CeO_2 slurry. By the developed CeO_2 grinding stone, a consumption of cerium oxide can be reduced to one fifth in comparison with a conventional polishing.

KEYWARDS : ceramic, cellulose, environment, cerium oxide particles, surface, hardness

INTRODUCTION

In general, a rigid magnetic memory disk loaded to a hard disk drive of a computer is based on an aluminium disk substrate and processed by fixed abrasives of a specific grinding stone[1]. The size of computer is becoming more compact and a ceramic disk or a glass disk substrate is becoming to be used as the memory medium in the field of compact size computer[2](smaller than 65mm diameter).

The magnetic disk substrate made from high purity aluminium alloy is used to be processed by a planetary type lapping machine to which a specific grinding stone is mounted, and accomplishes an excellent surface roughness, dimensional stability and also performs a good productivity. This method is accomplished by a newly developed resin bond grinding stone and a special improved operating technique for the planetary type lapping machine[1][3].

In the case of glass disk polishing, polishing by loose abrasives is commonly used. Fine particles of cerium oxide (CeO_2) having 0.5~2.5 micron meter average diameter are used as abrasives, which are supplied as the stabilized aqueous slurry to the surface of polisher. Polisher is the suede type non-woven cloth having a surface of fine void structure. By this method an excellent surface roughness can be obtained, however this method has problems in a productivity and an environmental pollution.

Fine particles of CeO_2, especially particles having very small diameter is very difficult to be immobilized as fixed abrasives in the matrix of grinding stone by conventional method for a preparation of grinding stone. It is said that the possible size to be immobilized is bigger than 3 micron meter.

In this paper, as the first step, a producing method of the grinding stone including CeO_2 abrasives is disclosed. The grinding stone in which cerium oxide fine abrasives are fixed is prepared by immobilizing fine particles in micro spherical cells made of cellulose and by fixing them into a matrix of resin bond grinding stone. As the second step, this paper refers to the experimental results of the grinding stone on a glass disk substrate.

PREPARATION OF CeO_2 GRINDING STONE

Polishing of Glass

In a case of the polishing of glass, by the effect of water a very thin hydration layer is formed on the surface of glass, and this hydration layer can be removed by the mechanical effect of CeO_2 abrasives which are supplied as the alkaline aqueous slurry[4]. Fine particles of CeO_2 abrasive acts as loose abrasives and rotate between the surface of glass and polisher, removes the thin hydration layer, thus the polishing is carried out.

Meanwhile, a precise processing by a grinding stone is becoming popular for the processing of aluminium substrate or silicon wafer, and is becoming to be used instead of a lapping or a polishing process. In a case of grinding, fine particles of abrasive are fixed in the matrix of grinding stone and does not rotate on the surface of glass during the grinding process. The sharpened edges of each fixed abrasives are pressed to the surface of work, and small plastic strain on the surface of work by shearing stress[2].

To obtain a mirror finished surface on glass by grinding stone grinding, it is necessary to fix fine particles of CeO_2 abrasive in the matrix of grinding stone.

Immobilization of CeO_2

Polyvinylalcohol resin has an excellent affinity as a specific bonding resin for various kinds of fine particles[5], and can be used as the base resin which composes the matrix of a grinding stone. However, the particles of CeO_2 to be used are too small to be immobilized in the matrix of grinding stone, even if the bonding resin is polyvinylalcohol resin. And also an aggregation problem of CeO_2 particles disturbs the fabrication of the good grinding stone. In general, it is said that the finest diameter of abrasives to be fixed in the matrix of a grinding stone is 1.2 micron meter.

The authors have carried out an earnest study, and arrived in our way of thinking to previously immobilize and enclose fine particles of CeO_2 into micro spherical cells as the first step. The average diameter of the micro spherical cell is 50-300 micron meter and includes fine particles of CeO_2 by high concentration. That is, the preparation of a micro complex spherical cell is the first step. Concretely the materials of the cell is cellulose[6]. The basic theory of this method is to immobilize previously fine particles of CeO_2 into micro spherical cells made by a specific material having an excellent hydrophilic feature and an affinity, and then the micro cells is fixed into matrix of grinding stone.

The role of the micro spherical cell is only to immobilize fine particles by high concentration, and the material of the cell must not give any effects to a physical feature of the grinding stone such as bonding strength, hardness or brittleness. Further, the material must be chemically and thermally stabilized. Cellulose is a material which is suited to the above requirements. Cellulose is a material which is prepared by treating pulp with a mixture of conc. NaOH and CS_2, has a lot of hydroxyl groups, and also an aqueous colloidal solution of cellulose (viscose) has an excellent dispersing ability for fine particles. Viscose has a good wetting and dispersing ability to fine particles which have wide surface area.

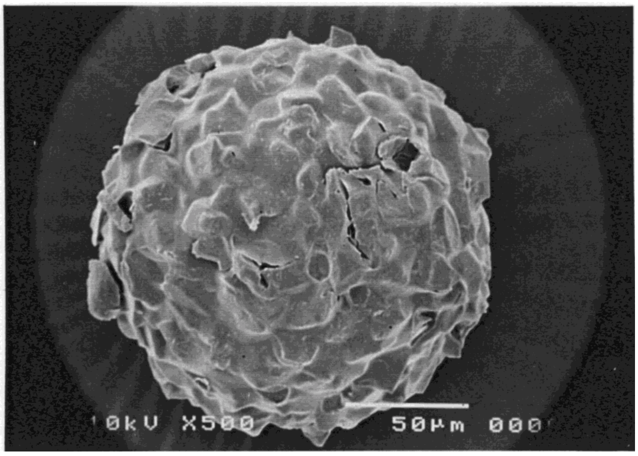

Fig.1 : SEM picture of the micro spherical cell with CeO_2 (·500)

A method to prepare the micro spherical cells of cellulose having a genuine spherical shape and to enclose fine particles of CeO_2 into it will be disclosed hereafter. Fig 1 is a microscopic picture illustrating the spherical cells of cellulose in which fine particles of CeO_2 having 0.1 micron meter diameter are immobilized. As clearly understood from the picture, the cells have a genuine spherical shape and the distribution of the diameter is closed to the state of single dispersion. The preparation of the micro spherical cells of cellulose is based on the phase separation phenomenon between aqueous solution of viscose and aqueous anionic solution of polymer. These two solutions do not have a good compatibility with each other, and when these two solution is mixed together cause a phenomenon of phase separation. Before the preparation, these two solution are adjusted as to be similar specific gravity and viscosity. Then they are mixed by desired ratio, stirred by constant speed and the temperature is gradually raised. By the constant stir, viscose is separated and forms fine and homogeneous liquid drops in aqueous anionic solution of polymer. And by the effect of temperature, viscose is solidified and cellulose is reclaimed. The reclaimed cellulose has a shape of fine spherical cell and has a soft and a jelly like feature. Then the obtained spherical cells are neutralized and dried up. By our study, it was found that the best material for aqueous anionic solution of polymer is the aqueous solution of sodium polyacrylate.

For the preparation of a micro cellulose spherical cells immobilizing fine particles of CeO_2 in it, above mentioned basic method is used. That is, fine particles of CeO_2 are thrown into a mixture of aqueous solution of viscose and aqueous anionic solution of polymer, then the particles of CeO_2 are transferred to the viscose phase which has a strong affinity to a fine particle. Thus, the micro cellulose spherical cells immobilizing fine particles of CeO_2 in it can be prepared. The average particle size of cells can be changed by the agitating speed[6].

Preparation of a grinding stone

Cellulose immobilizes fine particles of CeO_2, but the bonding strength to the particle is not so strong. And, since cellulose has a good affinity to various kinds of materials, the micro cellulose cell can be handled as if it is an independent particle of abrasive.

In this study, phenol resin is used as the bonding material together with polyvinylalcohol resin, and a wet manufacturing procedure is used for the preparation of the grinding stone which is quite different from that of a resinoid or a vitorified type grinding stone[1].

The aqueous solution of pre-polymer of phenol resin is mixed with the aqueous solution of polyvinylalcohol resin, then the necessary additives are added. The cells are thrown into the mixture, and the mixture is poured into a mold and placed into a water bath kept at 353K around temperature for about one day. Phenol resin and polyvinylalcohol resin are easily penetrated into the cellulose and immobilizes the fine particles of CeO_2 encapsulated in cells. The mixture is solidified by effects of a catalyst and a temperature and forms a phenol and polyvinylalcohol resin bond grinding stone. Fig 2 shows the SEM picture of micro cellulose spherical cells immobilizing and encapsulating fine particles of CeO_2 in it. The average size of cells is about 80 micron meter and concentration of CeO_2 is 80% by weight, and this type of micro cell is used in this study.

Before the main polishing test of glass, several kinds of grinding stone are prepared for trial and preliminary polishing test are carried out. For the preparation of grinding stone, abrasives of 2.2 micron meter size CeO_2 are used and only hardness (Young's modulus) is changed. Test results are shown in Table 1.

As clearly shown in Table 1, a harder type grinding stone does not have a cutting and polishing ability, and this result explains that the bonding resin is too hard to generate new abrasives on the stone surface. A softer type grinding stone has a problem in it's dimensional stability. During the polishing process it irregularly worn out by the friction, and the flatness of stone surface becomes wrong. In the extreme example, the stone surface become convex after polishing by the difference of friction caused by the difference of rotating velocity between inner portion and outer portion of plate.

Table 1 : Preliminary polishing test (abrasive size ; 2.2 micron)

grinding stone	No.1	No.2	No.3
abrasive conc. vol%	23.1	20.8	21.0
Rockwell hardness HRR	8.2	43.1	95.5
Young’s modulus MPa	2100	3850	5300

surface appearance	uneven	homogeneous	many scratches
surface roughness	partially good	mirror like	luster
stone surface	worn out	good	no change
stone wear	fast	normal	slow

From above mentioned preliminary polishing test, we assumed that the suitable physical features of grinding stone are,

Rockwell hardness (HRR) : 40-60

Bonding strength (mm/100) : 20-40

Young's modulus (MPa) : 3500-4500

Concentration of CeO_2 (vol%) : bigger than 20

POLISHING TEST OF GLASS

Preparation of grinding Stone

Based on the above mentioned results, the grinding stones including three different size CeO_2 (having average diameter of 0.7, 1.5 and 2.2 micron meter) are prepared and applied for the actual polishing tests of a glass. And the spherical cells having 80 micron meter diameter and including 80wt% of CeO_2 are used.

The physical properties of developed grinding stone are summarized in Table 2.

Table 2 : Grinding stones and polishing test results

size of CeO_2 micron	0.7	1.5	2.2
abrasive conc. vol%	20.0	20.7	21.6
resin conc. vol%	38.6	39.6	35.4
porosity vol%	40.5	39.7	43.0
Rockwell hardness HRR	48.6	50.5	44.3
bonding strength mm/100	32.9	27.5	26.9
Young’s modulus MPa	4250	3850	3800

Apparatuses for Estimation and Conditions

The developed CeO_2 grinding stones are mounted to a polishing machine and polishing tests are carried out on a disk shaped glass.

A polishing machine and operating conditions are follows,

Polishing machine : EJ-3801 type (Japan Engis Co., Ltd.)

Polishing period : 60minutes

Pressure: 49KPa

Rotating speed of grinding stone : $60min^{-1}$

Flow rate of polishing fluid : $5.3/min.m^2$

Polishing fluid : water

As the work to be polished, 40mm flat glass disk plate specimen is used, which is previously processed by Al_2O_3 loose abrasives lapping. Surface roughness of the specimen is 0.30 micron meter Ra and 2.4 micron meter Rmax, and also has a good flatness.

Test results

Polishing tests are carried out on a soda-lime glass specimen, using above mentioned grinding stones, polishing machine and conditions. Before each polishing test, the surface flatness of the platen composed by grinding stone is corrected by a dressing carrier plate. Fig 2 shows the test results of obtained surface roughness, and Fig 3 shows the test results of removal rate. Appearances of polished surface are almost same to a mirror finished surface, no scratches and no other defects can be observed on the surface. The surface is transparent and almost similar to the polished surface by CeO_2 loose abrasives.

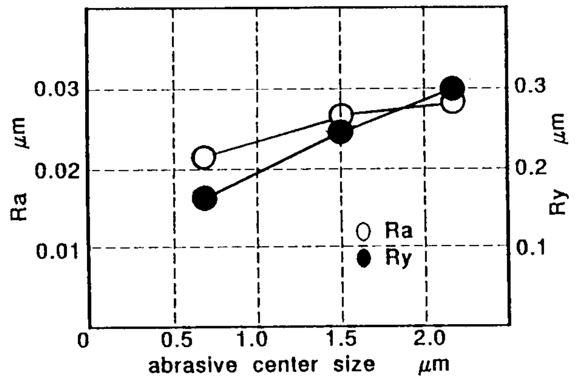

Fig.2 : Relationship between CeO_2 size and surface roughness

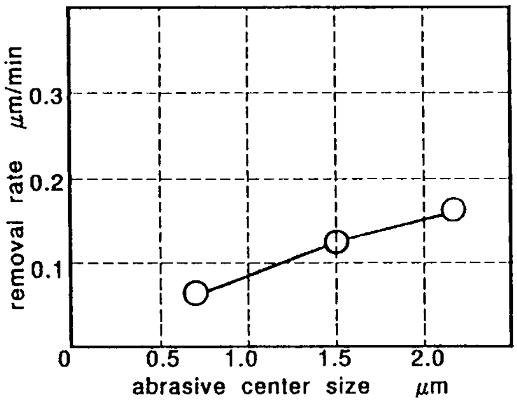

Fig.3 : Relationship between CeO_2 size and removal rate

As clearly understood from Fig 2 and 3, the surface roughness is satisfactory, however, the removal rate is too slow. Compared with that of the conventional polishing, the rate is 1/2 to 1/3 level.

To increase the removal rate, the tests to supply a small amount of CeO_2 slurry intermittently are made. Compared with the conventional polishing process, the total supply (consumption) of slurry is about 1/10. The obtained result are summarized in Fig 4 and 5. As clearly understood from these graphs, by the supplement of small amount of CeO_2 the removal rate is remarkably improved without affecting the surface roughness and other surface quality such as brightness and appearance.

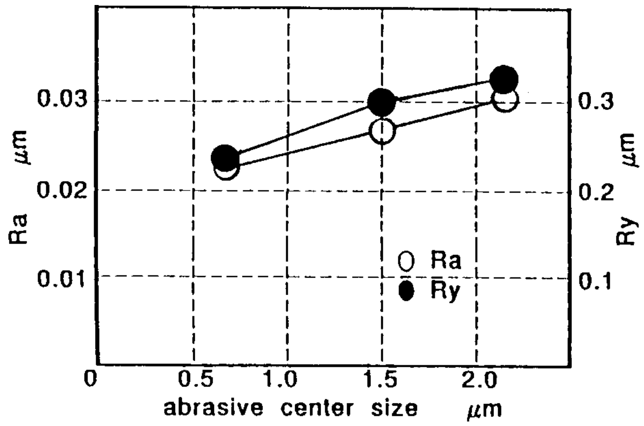

Fig.4 : Improvement of surface roughness by CeO_2 intermittent supply

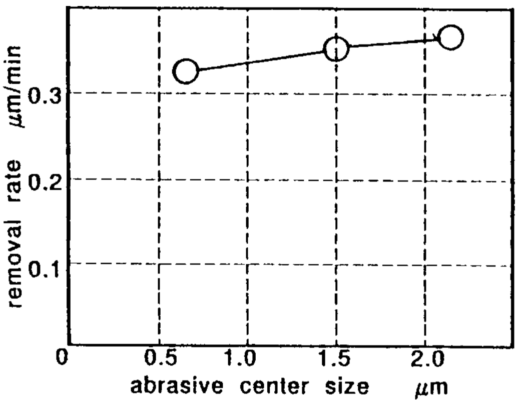

Fig.5 : Improvement of removal rate by CeO_2 intermittent supply

In general, in a case of the grinding by a grinding stone, obtained surface roughness is coarser than that of by a conventional lapping process[7]. However, in this paper almost same results are obtained on surface roughness from two different methods. This phenomenon will be discussed in detail in Discussion.

DISCUSSION

Developed new fixing method

Fine particles of CeO_2 are used as the abrasives for glass surface polishing. By contacting with water, a thin hydration layer is formed on the surface of glass. Fine particles of CeO_2 are used to remove the thin hydration layer so as to perform a mirror finished surface. Only particles of CeO_2 can remove said layer quickly without leaving scratches on the surface.

As a method to prepare a CeO_2 grinding stone, the method to solidify CeO_2 particles by high pressure in the presence of small amount of water has been reported [3].

The concentration of CeO_2 of the grinding stone by this method is very high (about 80vol%). On the other hand, the grinding stone of this paper has a lower concentration of CeO_2, and physical property is quite different.

For the preparation of a grinding stone, fine particles of CeO_2 must be mixed with various kind of chemicals. However, since CeO_2 contains a lot of impurity and is chemically unstable, when it is mixed with other chemicals the problems such as bubbling, denaturation, solidification or falling off come out and hurt the preparation of good grinding stone. Viscose is a chemically stable aqueous solution and electro statically neutralized, fine particles of CeO_2 can be easily immobilized in it without causing above mentioned problem and forms a spherical cellulose cell. Only by means of cellulose immobilization, CeO_2 can be fixed in the matrix of grinding stone, and this method can be estimated as the method to provide a new possibility of grinding stone containing abrasives of submicron grade.

Polishing test

Usually, glass polishing is carried out by using aqueous slurry of CeO_2 and polisher which is stuck on the iron plate of polishing machine. The suede type non-woven cloth having a fine void structure is used as a polisher. At the actual polishing, the aqueous solution of CeO_2 is constantly supplied to the polisher surface. Particles of CeO_2 abrasives are held in fine pores of void structure and act effectively to remove a thin hydration layer formed on the surface of glass. By using the suede type polisher scattering of slurry is prevented, and the elasticity of polisher prevent the scratching of the glass surface.

As shown in Fig 2, 3, 4 and 5, removal rate can be increased by additional supplement of aqueous slurry of CeO_2, however, surface roughness of polished glass is not changed. Particles of CeO_2 of grinding stone act as fixed abrasives. And particles of CeO_2 which are additionally supplied as aqueous slurry acts as loose abrasives.

In a case of conventional lapping process, the obtained surface roughness is changed by the state of abrasives, that is, the roughness obtained by loose abrasives is remarkably coarser than that obtained by fixed abrasives. Particles of loose abrasive rotate on the surface of work and very small breakings are generated by the “rolling action" of particle which proceeds the processing. The obtained surface is coarser, but the removal rate is high. When particles are fixed in the matrix of grinding stone, the edge of a fixed abrasive causes small plastic strain by shearing stress[7] and a finer surface can be obtained. That is, the processing mechanism by loose abrasives and by fixed abrasives are basically different.

On the other hand, in a case of the glass polishing, processing mechanism is different from above mentioned methods. Glass surface is chemically reacted with water and forms a soft hydration layer. By polishing with CeO_2, the layer is removed mechanically and the surface become finer. Regardless whether particles of abrasive are fixed or not, they acts only to remove the hydration layer, therefore the surface roughness is mainly influenced by the condition to generate the hydration layer.

By our experiment, it is clarified that the total amount of CeO_2 influences the removal rate but does not influence the surface roughness, and the state of particles of abrasive whether they are fixed or loose does not influences the surface roughness. This fact explains that the polishing process of glass is not performed by small breakings or small plastic strain, but performed by means of a mechano-chemical action.

CONCLUSION

Usually, it is said that the polishing of glass by fixed abrasive is impossible, however, in this paper it is clarified that the polishing of glass by fixed abrasive become possible by using a new immobilizing method in micro spherical cells.

The results obtained by this paper are summarized as follows.

1) The spherical hybrid cell including fine particles of CeO_2 in high proportion is developed based on the preparing technique of cellulose micro cells.

2) The manufacturing of grinding stone including CeO_2 abrasives by higher proportion become possible by using the above mentioned spherical cellulose cells immobilizing fine particles of CeO_2.

3) The polishing test of glass by fixed CeO_2 is carried out by using above mentioned grinding stone, and can obtained almost similar surface roughness with results obtained by conventional polishing, however the removal rate is slow.

4) The shortage of removal rate can be recovered by supplying intermittently a small amount of CeO_2 slurry without affecting a surface roughness.

5) The fact mentioned above clearly illustrates that the polishing of glass surface is not carried out by small breakings or small plastic strain, but performed by means of a mechano-chemical action on a thin hydration layer formed on the surface of glass.

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