Preparation of a Polyimide Composite Filled with Corundum as a Kind of Precision Grinding Slice

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ABSTRACT: By using a two-step synthetic method, a novel grinding material, SiC and polyimide grinding slice, is prepared. This grinding slice can be used for mechanical planarization of hard and brittle materials, such as silicon, optical glass, advanced ceramics, specials metal, jewelry, disks etc. For observing the physical and chemical properties of the slice, scanning electron microscopy, FTIR spectroscopy, thermal gravimetric analysis, tensile strength, and elongation at break are used. All the experimental data indicate that the SiC/PI slice will be a very useful grinding material for obtaining a precision surface. J Appl Polym Sci 113: 3033–3037, 2009

Key words: polyimide; thermal properties; films; interfaces; imaging

With the rapid development of high technology in recent years, high temperature resistant, high quality, and precision mechanical planarization[1–4] tends to be widely used and intensively studied. Polishing with abrasive films is one of the predominant methods for mechanical planarization, especially for hard and brittle materials including glass surface, ceramic components, optical lenses, semiconductor wafers, special metals, and computer memory disks etc.[5–7] Nowadays, more and more studies concentrate on the polishing techniques of abrasives[8–10] such as diamond and alumina with their substrates of polyurethane, ultra-high molecular weight polyethylene etc.[5] However, few articles have reported that polyimide (PI) can be used as abrasives' substrates.

PI is one of the most promising thermally stable polymers,[11,12] which has excellent properties of innocuity, acid resistance, alkali resistance, cauterization resistance, strong mechanical strength, good heat resistance, high stability in vacuum, antiradiation, solvent resistance etc.[13,14] On the basis of these advantages, PI has been applied to automobile, spacecraft, printed circuits, and other high-tech fields.[15-18] Today, PI and its composites attract extensive attention in the field of grinding materials and began to play a leading role in the area of tribology worldwide.[19,20]

In this study, a novel precision grinding slice, which can be widely used in the field of polishing, has been prepared using PI and corundum which are well mixed with sufficient stir. This kind of grinding slice has many advantages such as: the abrasive studs in PI uniformly and firmly, the slice endures high temperature (around 500°C) and the slice changes its functions by adding different abrasives (including different sizes).

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EXPERIMENTS

Materials

98.0% *N*,*N*-dimethyl acetamide (DMAC) was used as solvent; 98.5% pyromellitic dianhydride (PMDA) was dried at 160°C about 30 min before use; 98.0% 4,4′-oxydianline (ODA) was dried at 120°C about 30 min before use. Corundum particles with the average sizes of 13, 6.5, and 3 μm were used as abrasive, respectively. The drying processes were carried out in a DZF—6020 vacuum drying cabinet with its capacity of 2 L, temperature ranging from 10 to 200°C and degree of vacuum from 10^5 to 10^3 Pa provided by Qixin Scientific Equipment Co Limited.

Slice preparation

After drying process, same amount of ODA and PMDA were taken. ODA-DMAC solution was obtained by dissolving ODA into DMAC at the concentration of 0.5 mol/L with adequate stirring at room temperature. In an ice bath, PMDA was mixed into ODA-DMAC solution by adding very small amount each time while stirring vigorously. The ice bath was removed after all PMDA was added and the

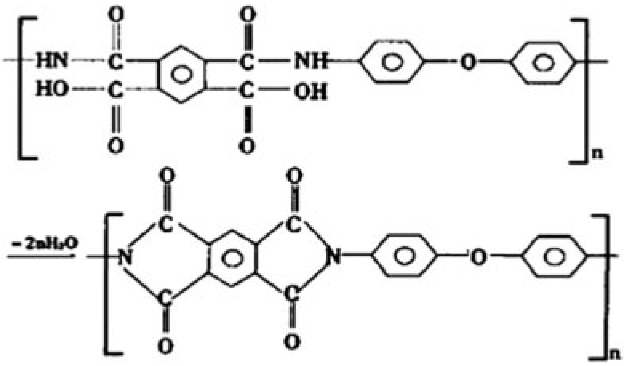

Figure 1 A schematic structure of transformation form PAA into PI.

solution was kept stirring heavily for 6–8 h. Then the mixture was heated to 75°C for 30 min with gentle stir. Heating was a necessary process through which a longer chained PAA could be obtained (Fig. 1).

Supersonic dispersion was applied to the solution of 1.2 g SiC in 30 mL DMAC. The solution was mixed with 0.5mol/L PAA until the total weight reached 100 g. A uniform mixture was obtained after the solution was vigorously stirred for 30 min. The mixture kept still around 10 min for eliminating the air bubbles, and then flow-casting method was applied by pouring and spreading the mixture onto a flat and leveled glass. During the drying process, the abrasive particles were aggregated at the bottom of the slice due to the gravity and a dual-layer film with a grinding layer (SiC/PI) and a glossing layer (PI) was obtained. Before the experiments, the dual-layer film was carefully designed since a stronger substrate, such as PI layer, was needed for supporting the grinding materials. A slice of evenly distributed SiC particles could also be obtained by changing the concentration/thickness of the DMAC and PAA solution for overcoming the gravitation. After drying in vacuum at 40–50°C for 24 h, the slice could be peeled off from the glass. The thermal imidization of SiC/PAA slice was carried out by a programmable increase of temperature from 25°C to 300°C and cooling down naturally in a muffle. Then a SiC/PI grinding slice was obtained.

The chemical structures of PI and PAA are described as follows: A SiC/PAA slice was formed by mixing SiC with PAA.

Measurements

The topography of grinding layer and the sectional structure were imaged by JSM-6380 scanning electron microscopy (SEM). The SEM was also applied to image the copper sheet surfaces which were grinded by the slices. Furthermore, at room temperature, the compositions of the grinding layer, glossing layer of SiC/PI grinding slice, and pure PI slice were analyzed by NICOLET380 FTIR spectroscopy (ATR, resolution 4 and scan 64). thermal gravimetric analysis (TGA) measurement was performed on an American TA SDTQ-600 DSC-TGA for 50 mg of the slice at the temperature from 25°C to 800°C with the increasing rate of 10 degree min^{-1}.The mechanical property was tested by Zwick/z020 Electronic Universal Testing Machine with its standard of GB1040-79. The sample size of the slice was 50×4 mm^2 in the shape of dumbbell and the testing distance was 18 mm at the rate of 50 mm/min.

RESULTS AND DISCUSSION

Topography of the slice

The dual-layer slice was imaged using SEM from top and side view. The images were expected to illustrate evenly distributed SiC particles on the surface and unevenly distributed in the bulk of the slice.

Figure 2(a) showed a clear dual-layer of SiC/PI and PI. The abrasive layer (at the bottom) was about 80 μm and the other was around 60 μm. Because of the gravitation, the heavy SiC particles aggregated at

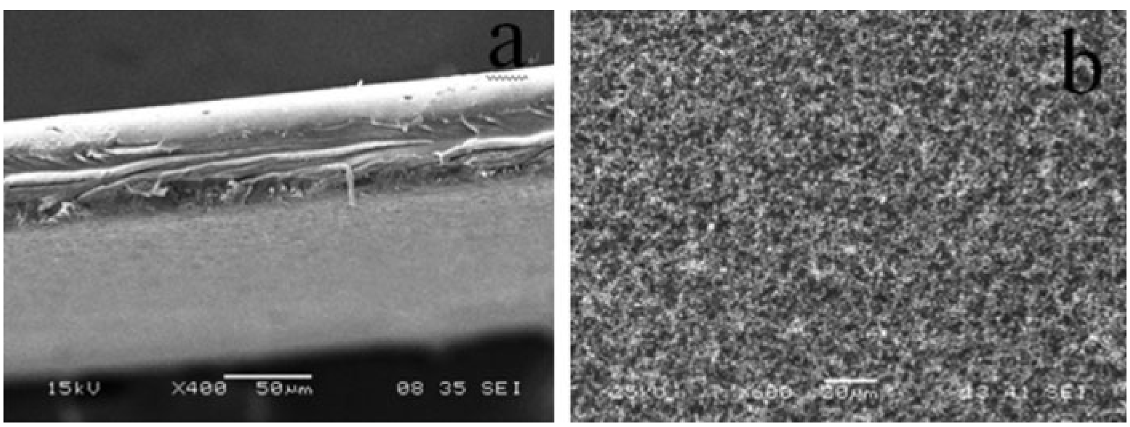

Figure 2 SEM images showed the particle size of 3 μm SiC /PI grinding slice (a, section view; b, top view).

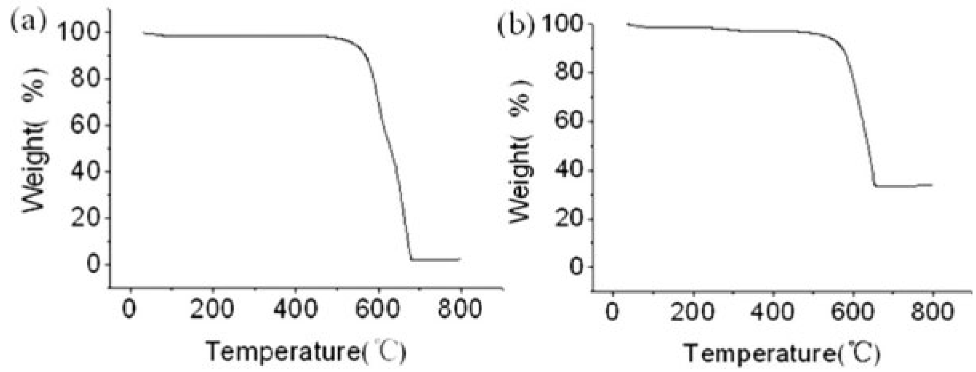

Figure 3 (a) TGA of pure PI slice; (b) TGA of 6.5μm SiC/PI grinding slice.

the bottom of PAA solution while drying and formed the abrasive layer. It could be seen clearly that the abrasive particles were spread evenly in the network of PI which ensured the stability of the particles while grinding. Figure 2(a) also showed a totally different PI structure (upper layer or glossing layer) which could be a supporting substrate for its superior thermal and mechanical properties (further discussion in later sections). Figure 2(b) was the top view of the grinding layer surface from which abrasive particles could be seen evenly studded in the slice surface.

Details were very important in the whole process of SiC/PI grinding slice preparation, which might have strong effects to the quality and properties of the slice. The original materials, like ODA and PMDA, should be dried thoroughly. PMDA was easily hydrolyzed resulting in the formation of *ortho*-diacid which could not only affect the molecular weight of PAA but also stop reactions with amic at low temperatures. A series experimental results showed that the PAA optimization concentration was about 0.5 mol/L. When higher concentration was applied, it was quite difficult to remove the air bubbles in the solution forming some defects on the slice. For lower concentration, however, the film would be too thin to use. In addition, the glass must be cleaned thoroughly with water and acetone, in turn, otherwise the organic remains absorbed on the glass could destroy the slice by pinholes.

FTIR spectra

The FTIR spectra for grinding layer and glossing layer showed the same characteristic peaks above 822 cm^{-1} as that of pure PI: 1776 cm^{-1}, symmetric C=O; 1720 cm^{-1}, asymmetric C=O; 1376 cm^{-1}, C—N stretch; 1500 cm^{-1}, aromatic ring. Peaks at 710 cm^{-1} however, the spectra showed big differences between grinding layer and glossing layer indicating the existence of SiC. Whereas, the glossing layer kept the same spectra as that of pure PI. The whole data of the spectra showed that no new chemical bond was formed during the slice preparing process implying the physical connection between PI and SiC. Thus, both SiC and PI will keep their own properties respectively, including hardness, heat resistance, acid resistance, alkali resistance, cauterization resistance, mechanical properties etc. However, the physical connection also indicated that the mechanical properties of SiC/PI layer were not as strong as that of PI parts.

Thermal gravimetric analysis

For observing the difference of disassemble temperature with and without SiC particles in PI slice, the measurements of TGA were carried out.

As shown in Figure 3(a), pure PI started to disassemble at 500°C and entirely finished the process at 670°C. Under the same temperature conditions, the SiC/PI grinding slice also disassembled at 500°C, however, it stopped losing weight at around 30% remains left which could be clearly seen by comparing Figure 3 (a) with (b). The remains should be corundum since its disassemble temperature is over 2600°C. Figure 3 also indicates that the SiC/PI slice has excellent thermal stability, around 500°C, which is superior to any other polymer based grinding slices.

TABLE I
The Tensile Strength and Elongation at Break of SiC/PI (SiC size ~ 13 μm)

	1	2	3	4	5	Average
Tensile strength/MPa	88.98	89.10	89.11	90.02	90.24	89.49
Elongation of break/%	20.33	20.04	21.29	22.44	22.51	21.32

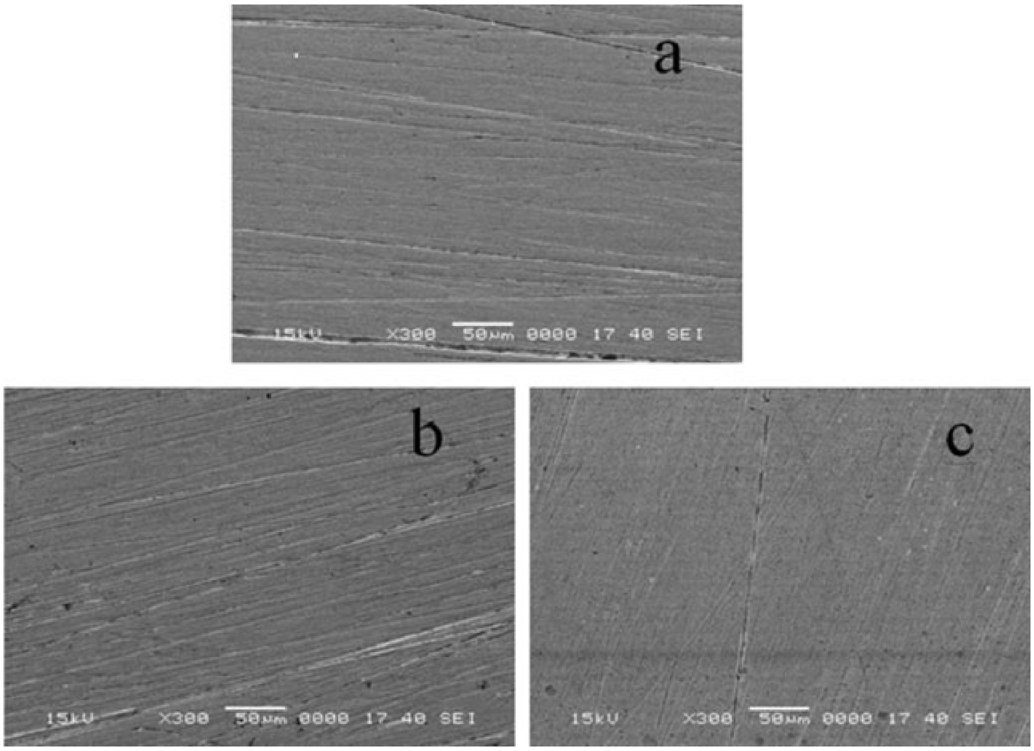

Figure 4 SEM images of copper sheet surfaces polished by SiC/PI slice with SiC particle size of (a) ~ 13 μm; (b) ~ 6.5 μm; and (c) ~ 3 μm.

Tension test

As a grinding material, a key factor of the slice is its mechanical properties. Thus, measurements of tensile strength and elongation of break were performed.

The mechanical properties of five pieces of SiC/PI dual-layer grinding slices were measured as shown in Table I. The average tensile stress and elongation at break were 89.49 MPa and 21.32%, respectively which were a bit lower than those of pure PI slices of 97.52 MPa and 34.64%. Even though the existence of mechanical reductions caused by the involvement of inorganic material SiC, the SiC/PI slices still showed excellent mechanical properties for heavy duty polishing tasks.

For comparing the mechanical properties of dual-layer structure, a single layer film was also prepared and the SiC/PI structure was formed evenly throughout the whole slice with its abrasive materials uniformly distributed by controlling the concentration of DMAC and PAA solution. For the same thickness of the slice, the mechanical strength of the dual-layer structure was better than that of a single layer film.

Polishing property of SiC/PI grinding slice

After polishing, the surfaces of three copper sheets looked very shiny and smooth with naked eyes. However, the SEM images showed great many scratches on their surfaces.

As shown in a–c of Figure 4, the deepest nicks could be found on the largest abrasive particles 13 μm grinded copper sheet surface and the lowest on the 3 μm grinded surface. The surfaces appeared smoother, flatter, and shinier when finer particles were applied. Therefore, a better surface could be obtained by changing the size of abrasive particles depending on the needs of precision.

CONCLUSIONS

A novel grinding material, SiC/PI grinding slice, has been synthesized which can be applied for precision metal, silicon, optical glass, and some advanced ceramics. The dual-layer slice, imaged by SEM, shows the SiC/PI layer is about 80 μm in thickness and the pure PI layer is about 60 μm. FTIR spectra analysis showed that the bonds between PI and SiC of the grinding slice are physical bonds which are not as strong as those of a pure PI slice. This may more or less affect the mechanical properties of the grinding slice. The tensile strength and elongation at break measurements indicate that SiC/PI grinding slice also have excellent mechanical properties. TGA results show that the SiC/PI grinding slice is thermally stable at the temperature below 500°C which means that the slice can undertake high temperature grinding tasks. This property is much superior to any grinding slice based on the substrates of normal polymer. SEM images of polishing experiments give us the hints the finer the abrasive particles, the more precision surface obtained. In consideration of its uniform distribution, hardness, heat resistance, acid resistance, alkali resistance, cauterization resistance, excellent mechanical properties, SiC/PI slice will be a very promising grinding material.

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