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Tribochemically Assisted Wear of Silicon Nitride Ball

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Abstract

The response of silicon nitride balls to different chemical additives contained in a diamond slurry was studied in a high speed four-ball machine. It was found that there are two effective inorganic chemical additives: acid additive (A) and alkaline additive (B). Use of A or B in the diamond slurry resulted in a high material removal rate and a rough surface. With an increase in the size of the diamond particles contained in the slurry, the material removal rate and the roughness of the surface also increased. Addition of sulfur-containing additives into the diamond slurries produced a decrease in the material removal rate but a smoother surface. The presence of water in the oil-based diamond slurry gave an increase in the material removal rate and a relatively smooth surface. In the ester-containing slurry, there was an increase in the material removal rate and the roughness of the surface.

1 Introduction

The in-service benefits of using advanced engineering ceramics (good chemical inertness, hardness, high temperature stability, low conduction of heat and high wear resistance) have led to their increasing use in industry. Unfortunately, engineering ceramics are very susceptible to surface damage during machining and surface finish operations, which affects their subsequent behaviour during service. To avoid damage during machining operations, the conventional grinding of engineering ceramics, for example, is a slow and expensive process, which often accounts for a significant portion of the total cost. Typically, surface finishing of ceramic components for high contact stress applications constitutes about 50% of the total cost of manufacturing.

With a continuous increase in the demand for advanced engineering ceramics, interest in their surface finishing processes has been steadily growing. Many modern surface finishing processes have been developed, such as magnetic force-assisted grinding, creep-feed grinding, ultrasonic-assisted grinding and energy-beam assisted grinding.[1,2] However, it seems that the dominant and the most effective industrial practice for the surface finishing of engineering ceramics is still a grinding process using diamond slurries. To understand the grinding process and the factors affecting high material removal rates and controlling surface damage, a comprehensive study into the optimum conditions of grinding silicon nitride balls in diamond slurries was carried out.[3]

Nowadays, increasing attention is being paid to the potential of using tribochemical effects during surface finishing of engineering ceramics.[4] Based on experimental results of adding many kinds of additives into the base oil, Gates and Hsu[5] produced a list of prowear and antiwear effects of the additives during sliding of engineering ceramics under lubricated conditions. Later, Wang and Hsu[6] reported an increase in the material removal rate during chemically assisted machining of engineering ceramics. However, it appears that there is a need to understand the mechanism of the process taking place during grinding of engineering ceramics in the diamond slurry, so that the effectiveness of tribochemistry in the surface finishing of brittle materials can be maximized.

In this paper, the results of a study on the chemically assisted grinding of silicon nitride balls are presented. The work concentrates on understanding of tribochemical effects occurring during grinding in diamond slurries containing different chemical additives, in order to develop more effective grinding processes of engineering ceramics.

2 Experimental Procedure

2.1 Apparatus

All experiments presented in this paper were

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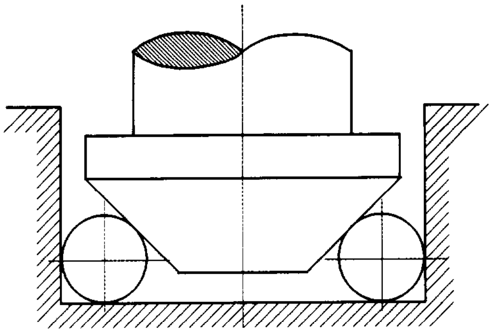

Fig. 1. Schematic diagram of the three-point configuration used for the grinding experiment.

carried out on a model contact configuration. In the normal configuration, an upper ball is located in the collet carried by the vertically mounted spindle. Three lower balls are allowed to rotate freely in a special race when brought into loaded contact with the single upper ball. In the study reported here, the configuration was modified as shown in Fig. 1. The upper ball was replaced by a metal cone with an angle of 90° and the three lower balls were replaced by nine silicon nitride balls with 6·5 mm diameter. The balls were free to rotate inside a metal cup. The cone together with the spindle was driven by an electronically controlled motor. In this case, each ball was in contact with the cup at two points and with the cone at one point. For this reason, the configuration was called 'three-point contact'.

2.2 Materials

The silicon nitride balls with 6·5 mm diameter were manufactured by the method of hot-isostatic pressing. The cone and cup were fabricated from 304 grade stainless steel.

In this study, two types of grinding liquid were used: base diamond slurry and 'tribochemical' slurry. The base diamond slurry contained diamond particles of different size. They were 1 μm or 15 μm oil-based diamond slurries (O-type) and 1 μm or 15 μm water-based diamond slurries (W-type). The seven variations of tribochemical slurry used are characterized in Table 1.

Two types of chemical additive were used: inorganic additives and organic additives. The inorganic additives were of an acid type (additive A) and an alkaline type (additive B). The organic additives are listed in Table 2.

To prepare a grinding liquid with tribochemical action, 15 μm diamond particles were suspended in the tribochemical slurry. The concentration of diamond particles was 2 wt%. Seven organic additives were added into the O-type oil-based diamond slurries in different weight concentrations. The inorganic additives were added to the pre-mixed diamond slurry with 50% W-type (water-based) and 50% O-type (oil-based) diamond slurries (WODS).

2.3 Experiments

A typical experiment began by placing silicon nitride balls on the pre-run track in the stainless steel cup and submerging them in about 4 ml of diamond slurry (1 or 15 μm diamond particle size). The load of 400 N was carefully applied to the set of nine silicon nitride balls. The test was

Table 1. Properties of the tribochemical slurries

Slurry	*Composition of additive*	*Pour point (°C)*	*Colour*	*Viscosity at 40°C (cSt)*	*Flash point (°C)*
853	Highly refined mineral oil	< 18	amber	19·0	190
854	Highly refined mineral oil	< 9	amber	30·0	200
855	Ester basestocks	< 20	amber	27–31	180
856	Poly-alpha-olefin	—	colourless	5	135
857	Poly-alpha-olefin	< 68	colourless	31	200
858	Polyoxyalkylene glycol ether	< 25	yellow	180	180
884	Ester basestocks	< 20	amber	85	180

Table 2. Properties of the organic additives

Additive	*Composition*	*Odour*	*Water solubility*	*Density (kg m^{-3})*	*Flash point (°C)*	*Viscosity at 40°C (cSt.)*	*Viscosity at 100°C (cSt)*
995	Ca sulfonate	mild	insoluble	1220	160	1200	60
996	Na sulfonate	mild	partially soluble	—	160	—	200
997	Olefin sulfide	mild	insoluble	1045	>100	45	6
998	Olefin ester sulfide	strong	insoluble	960	190	300	35
HT312	Sulfide	mild	insoluble	1160	95	—	9·5
DBDS	Dibenzyldisulfide	mild	—	—	—	—	—
NSE	Natural sulfurized ester	strong	—	—	—	—	—

carried out at the 3000 rev min^{-1} rotational speed of the spindle and lasted for 1 h.

The silicon nitride blank balls, in the as-received condition, had rough surfaces of the order of 100–200 μm R_a and contained surface imperfections. Therefore, they were subjected to a run-in process before the grinding test to remove the rough surface layer and imperfections. Running-in was carried out in the two steps. Firstly, 0·5 g of 15 μm diamond paste was smeared on the set of blank silicon nitride balls and 4 ml of a 15 μm oil-based diamond slurry was added into the stainless steel cup. The set of nine blank balls was pre-run at 100 N and 700 rev min^{-1} speed for 1 h. Then, the load was increased to 200 N and the speed increased to 3000 rev min^{-1}. Under this condition, the silicon nitride balls were run-in for 2 h. During this process, approximately 300 μm of silicon nitride was removed from each ball, which resulted in the average reduction of their diameters from the initial 6·8 mm to 6·5 mm.

After the running-in of the silicon nitride balls, they were ready for the test (i.e. ~6·5 mm diameter). Before and after the tests, all silicon nitride balls and the stainless steel cup were ultrasonically cleaned thoroughly in solvents. The material removal was assessed by the decrease in diameter of the silicon nitride balls after the grinding test. The material removal rate per unit time was calculated. Scanning electron microscopy (SEM) was used to obtain micrographs of the surfaces of the silicon nitride balls at low and high magnifications before and after the grinding tests under the different conditions.

3 Results

3.1 Material removal rate in the presence of water

Grinding tests were carried out in WODS. Figure 2 shows how the water affects the material removal rate of the silicon nitride balls during grinding in the diamond slurry: an improvement (~30% increase) in the material removal rate of the silicon nitride balls was achieved during grinding in the WODS comparing with grinding in the oil-based diamond slurry.

3.2 Material removal rate in the presence of inorganic additives

3.2.1 Different inorganic additives

To examine the effect of inorganic additives with different chemical characteristics, two kinds of inorganic additives, i.e. acid additive (A) and alkline additive (B), were used. They were added into the pre-mixed diamond slurry consisting of 50% water-based and 50% oil-based diamond slurries (WODS). Figure 2 shows the material removal rate after grinding silicon nitride balls in the 1 μm oil-based diamond slurry, 1 μm WODS, 5 mol dm^{-3} of A in 1 μm WODS and 5 mol dm^{-3} of B in 1 μm WODS. It is seen from Fig. 2 that the lowest material removal rate took place during grinding in the oil-based diamond slurry. Adding 5 mol dm^{-3} of A or B into 1 μm WODS resulted in an increase of the material removal rate. The increase is greater than sixfold and greater than fourfold, respectively, for 5 mol dm^{-3} of A and B in 1 μm WODS.

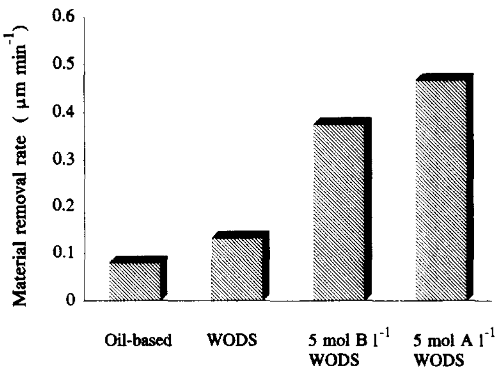

Fig. 2. Effect of water and inorganic additives on the material removal rate of silicon nitride balls during grinding at 400 N load and 3000 rev min^{-1} speed in 1 μm WODS.

3.2.2 Concentration of inorganic additives

The effect of different concentrations of additives A and B in 1 μm WODS on the material removal rate of silicon nitride balls was also studied. The results are given in Fig. 3. It is seen that the material removal rate increases with increasing concentration of both the additives. It appears from Fig. 3 that the initial dramatic increase in material removal rate stabilizes at a constant value even though a further increase in the concentration of additives takes place. Figure 3 also shows that the material removal rates were higher during grinding in WODS with the additive A than with the additive B throughout the whole range of concentrations.

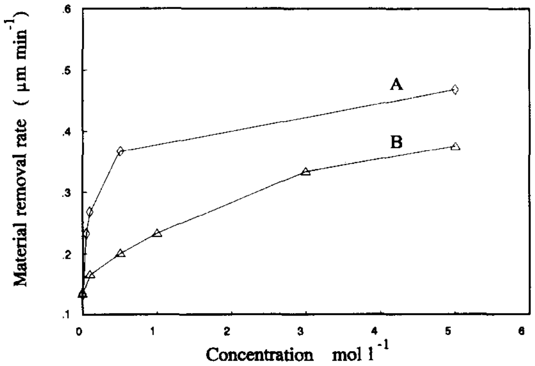

Fig. 3. Variation of the material removal rate of silicon nitride balls with the concentration of inorganic additives in 1 μm WODS during grinding at 400 N load and 3000 rev min^{-1} speed.

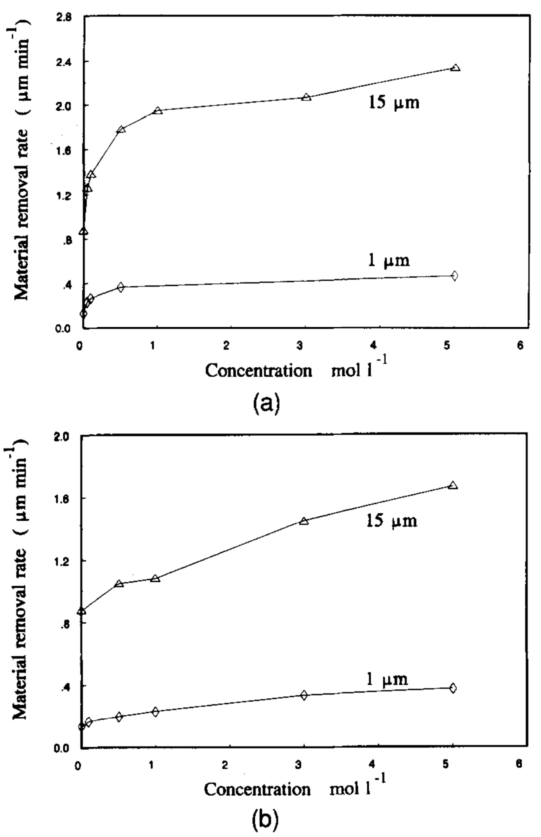

Fig. 4. Variation of the material removal rate of silicon nitride balls with the concentration of inorganic additive A (a) and inorganic additive B (b) in 1 μm and 15 μm WODS during grinding at 400 N load and 3000 rev min^{-1} speed.

3.2.3 Effect of diamond particle size

Inorganic additives were added to WODS containing diamond particles of different sizes. Figures 4(a) and (b) show the rate of material removal resulting from grinding in the 1 or 15 μm WODS with varying concentrations of additives A and B, respectively. It is seen that the material removal rate increases both with increasing size of the diamond particles and increasing concentration of inorganic additives. The material removal rate is over five times higher when grinding in the 15 μm slurry with the inorganic additive A or B than in the corresponding slurry containing 1 μm diamond particles.

As indicated earlier, there was an initial sharp increase in the material removal rate with increasing concentration of additive A. Afterwards the rate stabilized at a more or less constant value.

3.3 Material removal rate in the presence of organic additives

3.3.1 Different base tribochemical slurries

Tribochemical effects produced by organic additives during grinding of silicon nitride balls were studied by adding 15 μm diamond particles to a number of base tribochemical slurries. To compare the grinding behaviour, a reference slurry

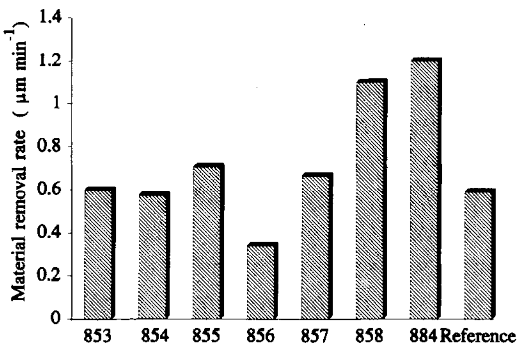

Fig. 5. Material removal rate of silicon nitride balls during grinding at 400 N load and 3000 rev min^{-1} speed in the tribochemical slurries with 15 μm diamond particles.

was produced by removing the diamond particles from the oil-based slurry and replacing them by the same 15 μm diamond particles used for base tribochemical slurries. This slurry was called the reference slurry. Figure 5 gives the material removal rates during grinding in the various tribochemical diamond slurries and reference slurry. It appears from Fig. 5 that the highest material removal rate was achieved during grinding in the 884 diamond slurry and the lowest in the 856 diamond slurry.

A high material removal rate also occurred during grinding in the 858 diamond slurry, as shown in Fig. 5. However, it was found that after grinding the silicon nitride balls were not completely spherical, the maximum difference in diameter being almost 10 μm. A difference in diameter was also found after grinding the silicon nitride balls in the 856 and 857 diamond slurries; this time the difference was 6 and 8 μm, respectively.

3.3.2 Base diamond slurry with organic additives

To study the effect of organic additives on the grinding behaviour of the silicon nitride balls, seven different additives were added into 1 μm oil-based diamond slurry at a concentration of 1 wt%. The results are shown in Fig. 6. It is seen that a decrease in the material removal rate occurred during grinding in HT312, dibenzyldisulfide (DBDS)

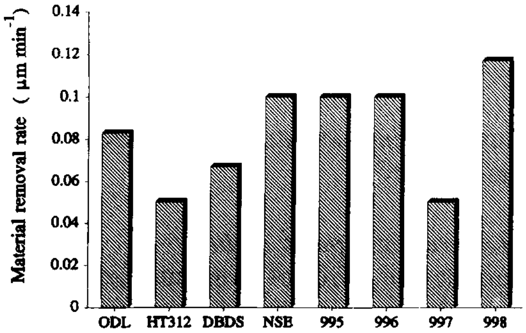

Fig. 6. Effect of organic additives on material removal rate of silicon nitride balls during the grinding at 400 N load and 3000 rev min^{-1} speed in 1 μm oil-based diamond slurry.

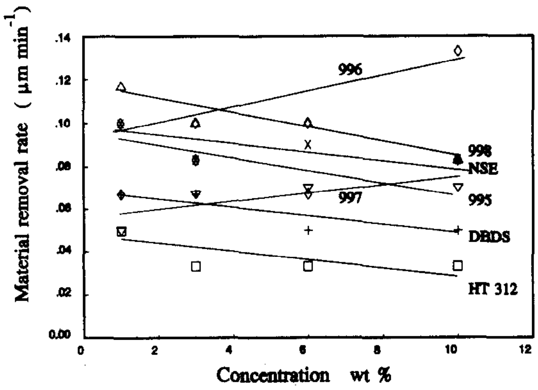

Fig. 7. Variation of the material removal rate of silicon nitride balls with concentration of organic additives in 1 μm diamond slurry during grinding at 400 N load and 3000 rev min^{-1} speed.

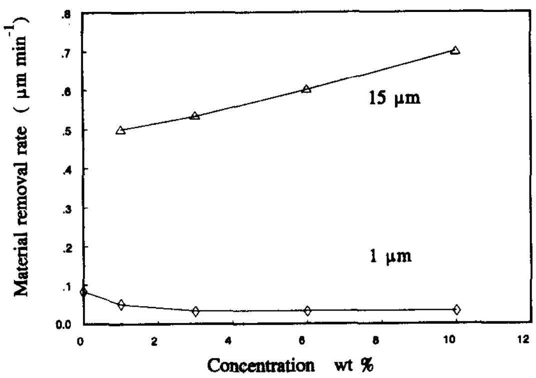

Fig. 8. Effect of the concentration of organic additives and the size of diamond particles in a diamond slurry on the material removal rate of silicon nitride balls during grinding at 400 N load and 3000 rev min^{-1} speed.

and 997 slurries. On the other hand, an increase occurred when natural sulfide ester (NSE), 995, 996 and 998 slurries were used.

The material removal rate as a function of organic additive concentration in 1 μm oil-based slurry was also examined. The results indicate that, during grinding in most of the diamond slurries, the material removal rate decreases with an increase in the concentration of organic additive (see Fig. 7). However, the reverse results were obtained using 996 and 997 organic additives, with the material removal rate increasing. Figure 8 gives the variation of the material removal rate with increasing concentration of the HT312 additive in 1 and 15 μm diamond slurries.

4 Discussion

4.1 Characteristics of ground surface in the presence of water

It was reported that, during sliding of silicon nitride in the presence of water, both friction and wear decrease.[7] In this study, in contrast, an increase in the material removal rate occurs during grinding of the silicon nitride in the oil-based diamond slurry containing water (see Fig. 2). Examining the surfaces after tests in both oil-based diamond slurry and WODS, it is evident from Fig. 9 that different surface appearances were produced during grinding. Both surfaces are covered with scratches on the micrometre scale, which indicates a ploughing action during grinding (see Figs 9(a) and (c)). At a high magnification, scratches on the nanometre scale can also be seen on the ground surfaces (see Figs 9(b) and (d)).

It is apparent from Fig. 9 that there are two major differences between both ground surfaces. All the scratches on the surface produced by the grinding test in 1 μm oil-based slurry have very clear edges (Fig. 9(a)). However, it seems that the scratches on the surface produced by the grinding test in WODS are shallower and smaller than those produced by the grinding test in 1 μm oil-based diamond slurry (see Fig. 9(c)). It is evident at a high magnification that the edges of the scratches are not sharp (see Fig. 9(d)). On the other hand, the number of the scratches decreased dramatically after grinding in 1 μm WODS, as shown in Figs 9(c) and (d). It can be postulated, therefore, that the surface produced by grinding in WODS is smoother than that in the oil-based diamond slurry. This is in agreement with the finding[4] that a smooth surface resulted from the tribochemical nature of the reaction of silicon nitride with water.

4.2 Characteristics of ground surface in the presence of inorganic additives

Addition of additive A or B to WODS caused a remarkable increase in the material removal rate, as shown in Figs 2 and 3. Examination of the ground surfaces reveals an uneven topography after the tests in 1 μm WODS with 5 mol dm^{-3} of A or B. This can be seen in Figs 10(a) and (b). These figures indicate that the high rate of material removal is accompanied by the formation of a relatively rough surface.

Closer examination of Fig. 10(a) reveals the existence of black areas on a white background under a scanning electron microscope. At high magnification, the black areas look very smooth, as can be seen in Fig. 11(a). The white areas are rough with an open surface texture and are considered to be pitted regions created by tribochemical action, as can be seen in Fig. 11(b). With decreasing concentration of the additive A in WODS, the percentage of the pitted regions in the total contact surface decreased. Finally, at the lowest concentration of A in 1 μm WODS equal to 0·05 mol dm^{-3}, the pitted regions were reduced to islands as shown in Fig. 12(a). More characteristic features

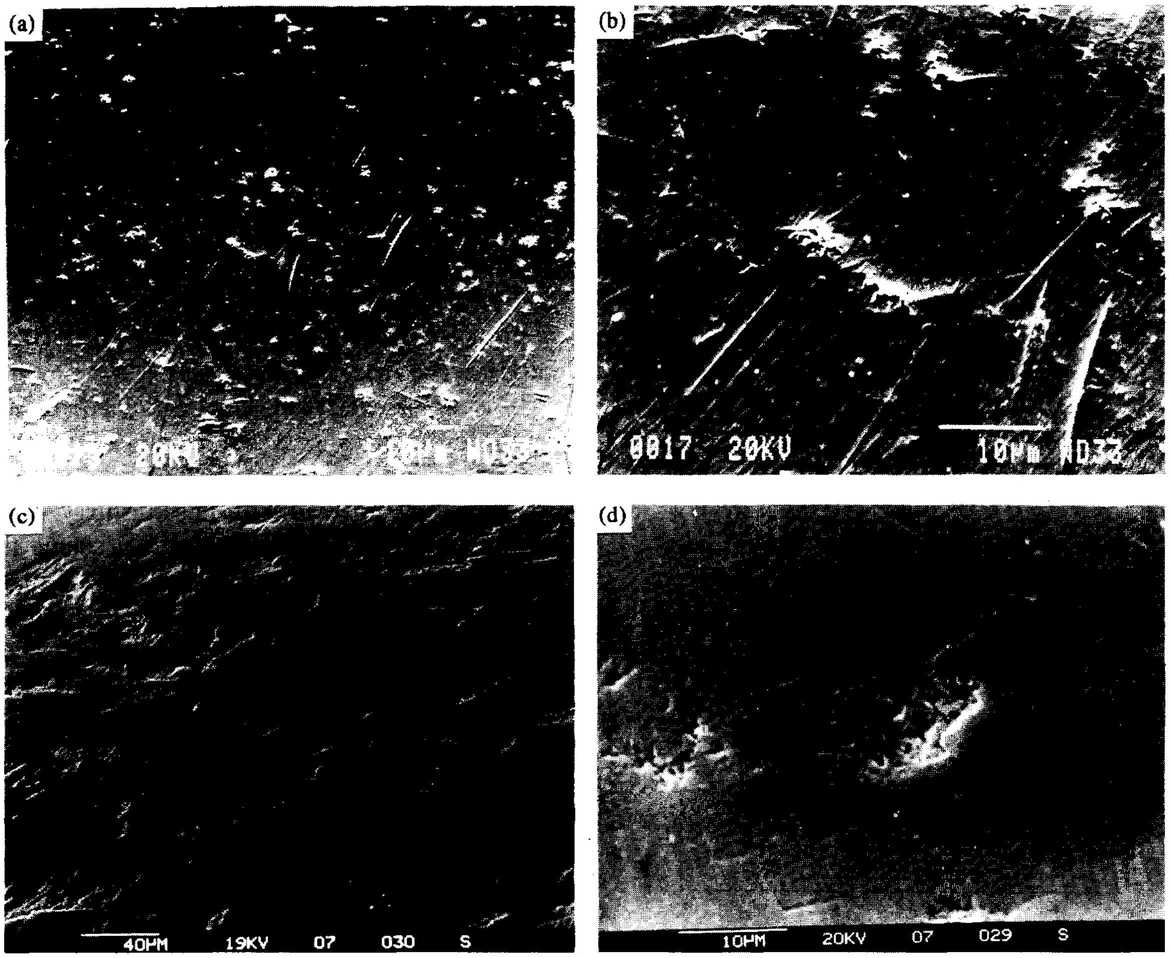

Fig. 9. SEM micrographs of ground surfaces of silicon nitride balls after grinding tests at 3000 rev min^{-1} speed and 400 N load in 1 μm oil diamond slurry (a and b) and 1 μm WODS (c and d).

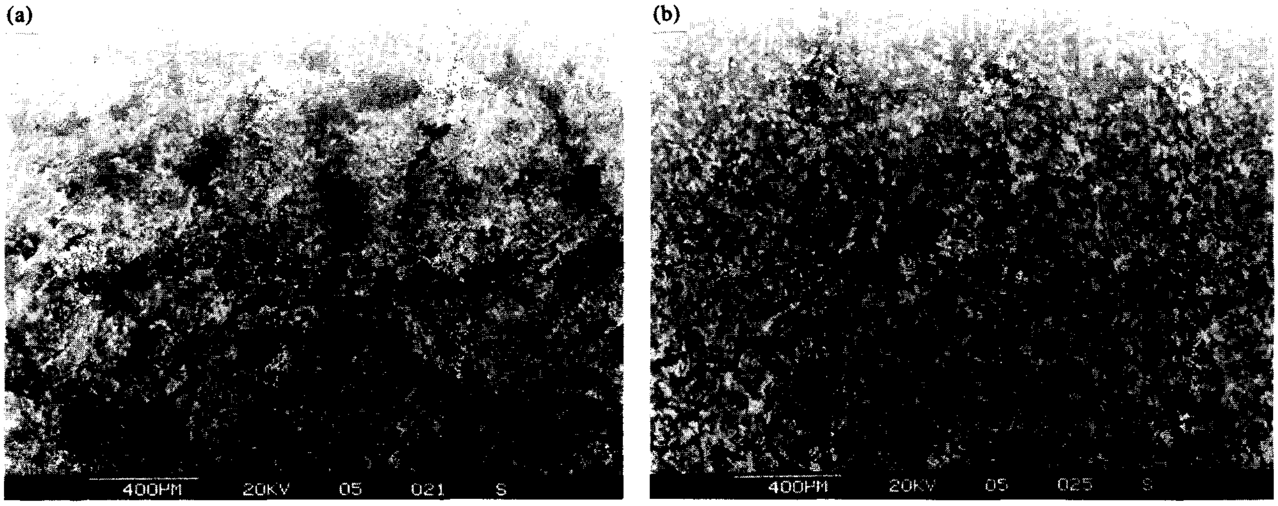

Fig. 10. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in (a) 5 mol dm^{-3} of A in 1 μm WODS and (b) 5 mol dm^{-3} of B in 1 μm WODS.

produced by grinding in 0·05 mol dm^{-3} of A in 1 μm WODS are shown in Figs 12(b) and (c).

Figure 10(b) reveals that there are many pitted regions produced by the grinding test in 5 mol dm^{-3} of B in 1 μm WODS. Examination of these regions at a high magnification indicates that they appear to be pits on a smooth black surface (see Fig. 13(a)). Like the pitted regions formed by the grinding test in 5 mol dm^{-3} of A in 1 μm WODS, the pitted regions show a rough surface with an open surface texture (see Fig. 13(b)).

4.3 Characteristics of ground surface in the presence of organic additives

The diamond slurries with organic additives included the blank, the sulfur-containing and the

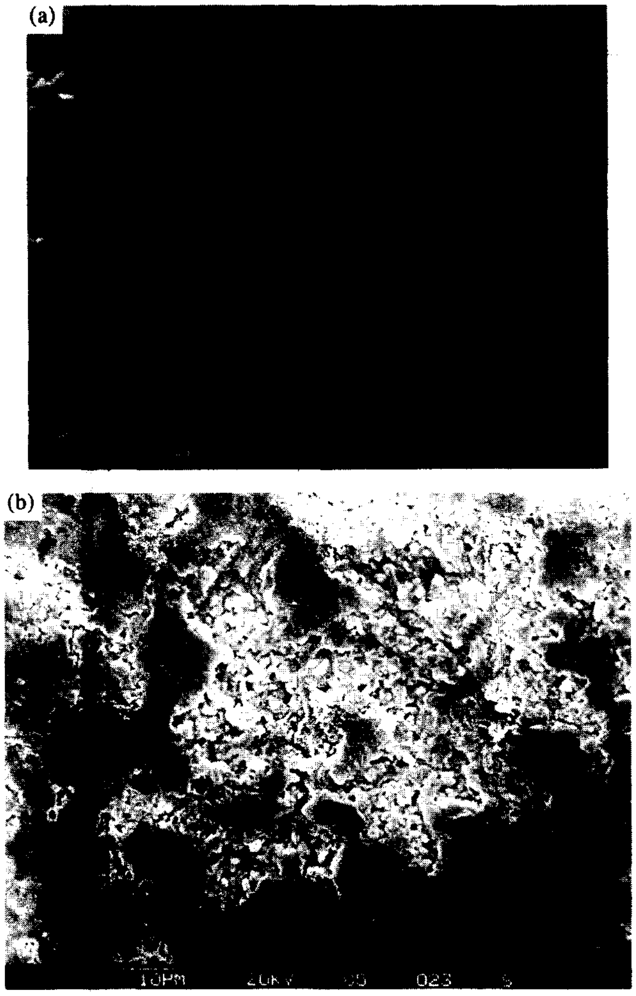

Fig. 11. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 5 mol dm^{-3} of A in 1 μm WODS. (a) Smooth black area, (b) rough pitted region.

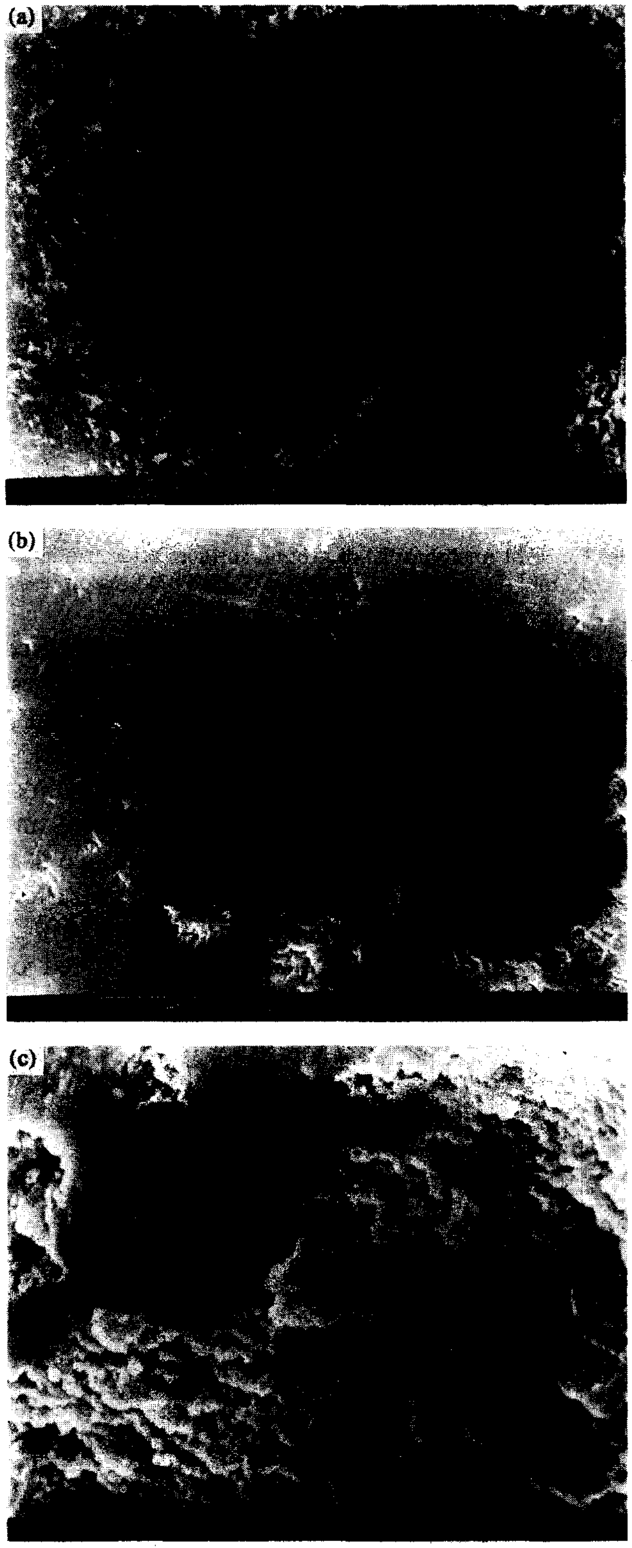

Fig. 12. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 0·05 mol dm^{-3} of A in 1 μm WODS. (a) Islands of the pitted region, (b) smooth black area, (c) rough pitted region.

ester-containing diamond slurries. The blank diamond slurries were those without an organic additive, and included the oil-based, the highly refined mineral oils 853 and 854, the poly-alpha-olefins 856 and 857, and polyoxyalkylene glycol ether 858. With the exception of the 858 slurry, all blank slurries gave a similar material removal rate, as shown in Fig. 5. However, the 856, 857 and 858 slurries caused ovality of the balls which is an undesired side-effect.

The sulfur-containing slurries included the HT312, the 997 and the DBDS diamond slurries. The experimental results indicated a decrease in the material removal rate during grinding tests in the sulfur-containing slurries due to the presence of the sulfur-containing additives (see Fig. 6). However, microscopic examination of ground surfaces revealed an excellent surface finish, as can be seen in Figs 14(a) and (c). Gates and Hsu[5] pointed out that the smooth surface of the silicon nitride results from the formation of a surface film during sliding in the sulfur-containing slurries. A study on the surface micro-topography at high magnification showed the existence of very fine scratches produced by micro-cutting after grinding tests in the sulfur-containing slurries, as shown in Figs 14(b) and (d). Some pitted regions a few micrometres in size were also found, as can be seen in Fig. 14(d).

The 995 and 996 additives are metal-containing sulfonates. The material removal rate was slightly higher during grinding with these additives than in the blank oil-based diamond slurry (see Fig. 6).

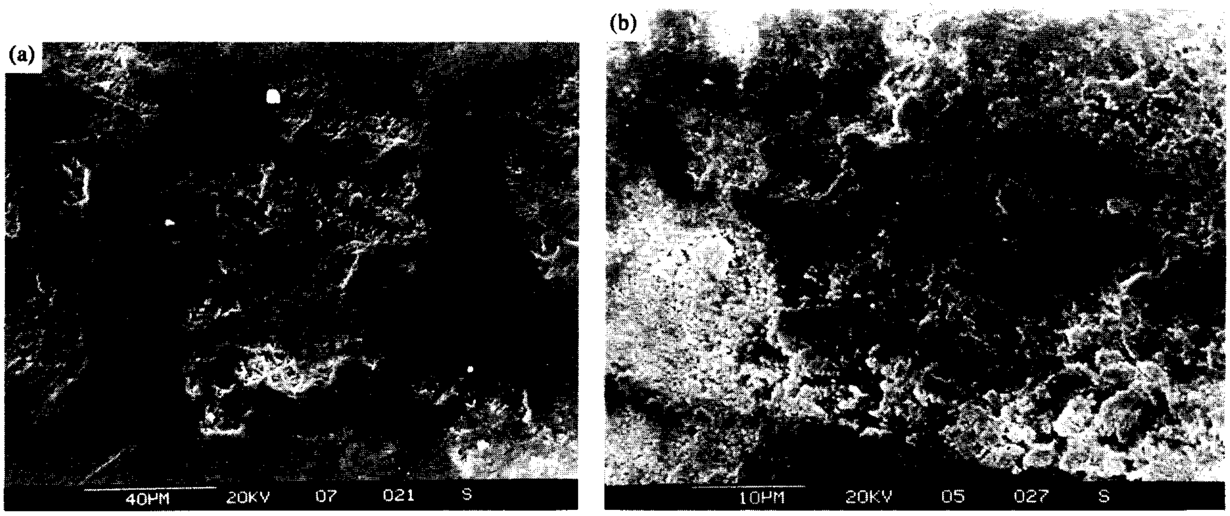

Fig. 13. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 5 mol dm^{-3} of B in 1 μm WODS. (a) Pitted region resembling a pit on the smooth black area, (b) rough pitted region.

It was found that the slurry viscosity increased upon the addition of the 995 and 996 additives. Figure 15 shows the ground surface of silicon nitride ball after the test in the 995 and 996 slurries. It is seen from Figs 15(a) and (c) that most regions on the ground surface show an excellent surface finish. Observation of the ground surface at a high magnification indicates the presence of a few deep grinding scratches (see Figs 15(b) and (d)).

Ester-containing diamond slurries consisted of the 855, the 884, the 998 and the NSE slurries. From Figs 5 and 6, it is apparent that grinding in the ester-containing slurries resulted in an increase in the material removal rate. Microscopic examination of the surfaces ground in the 998 and NSE slurries revealed fine scratches and pitted regions, as shown in Fig. 16. Both 998 and NSE contain sulfur, which may have contributed to the polishing process. However, the exact mechanism is not known.

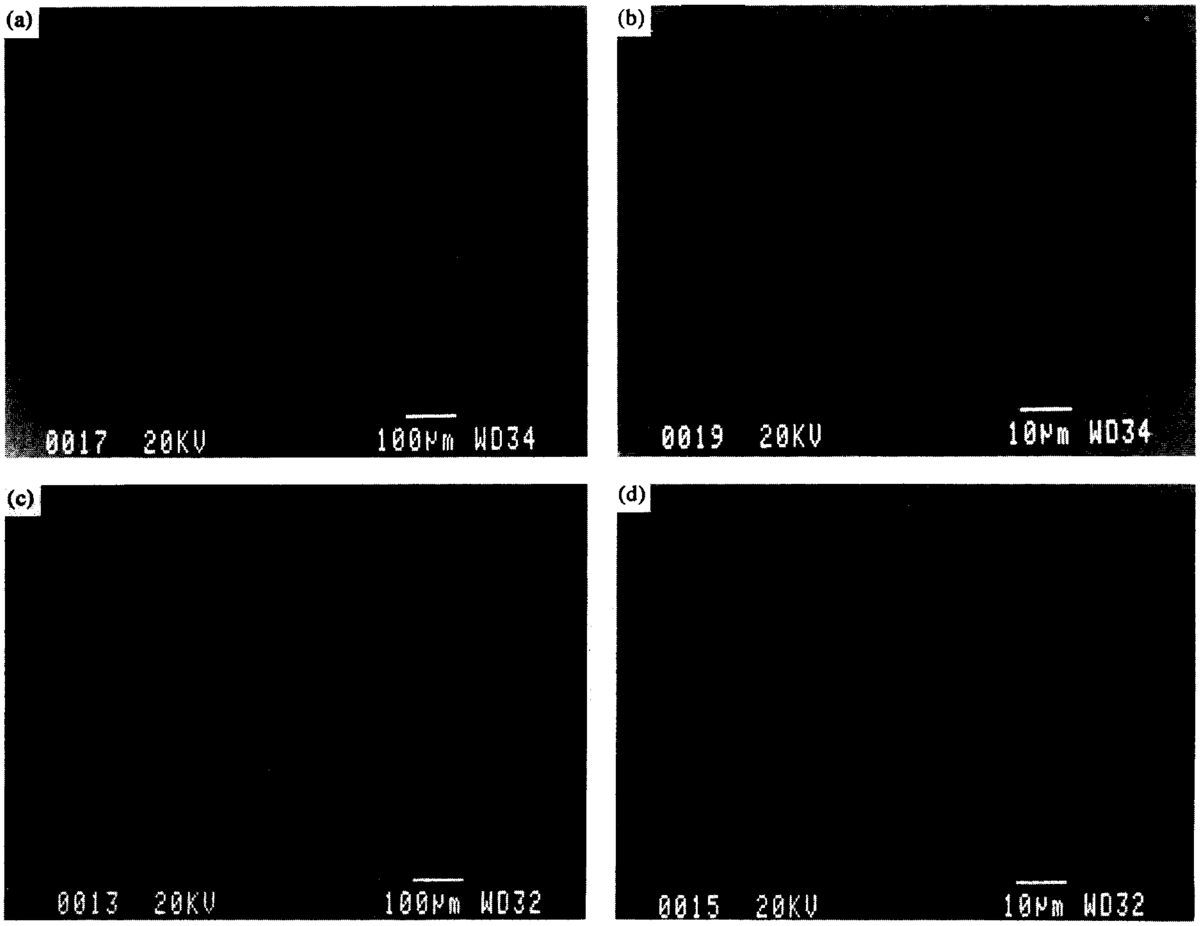

Fig. 14. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 1 wt% of 997 in 1 μm diamond slurry (a and b) and 1 wt% of DBDS in 1 μm diamond slurry (c and d).

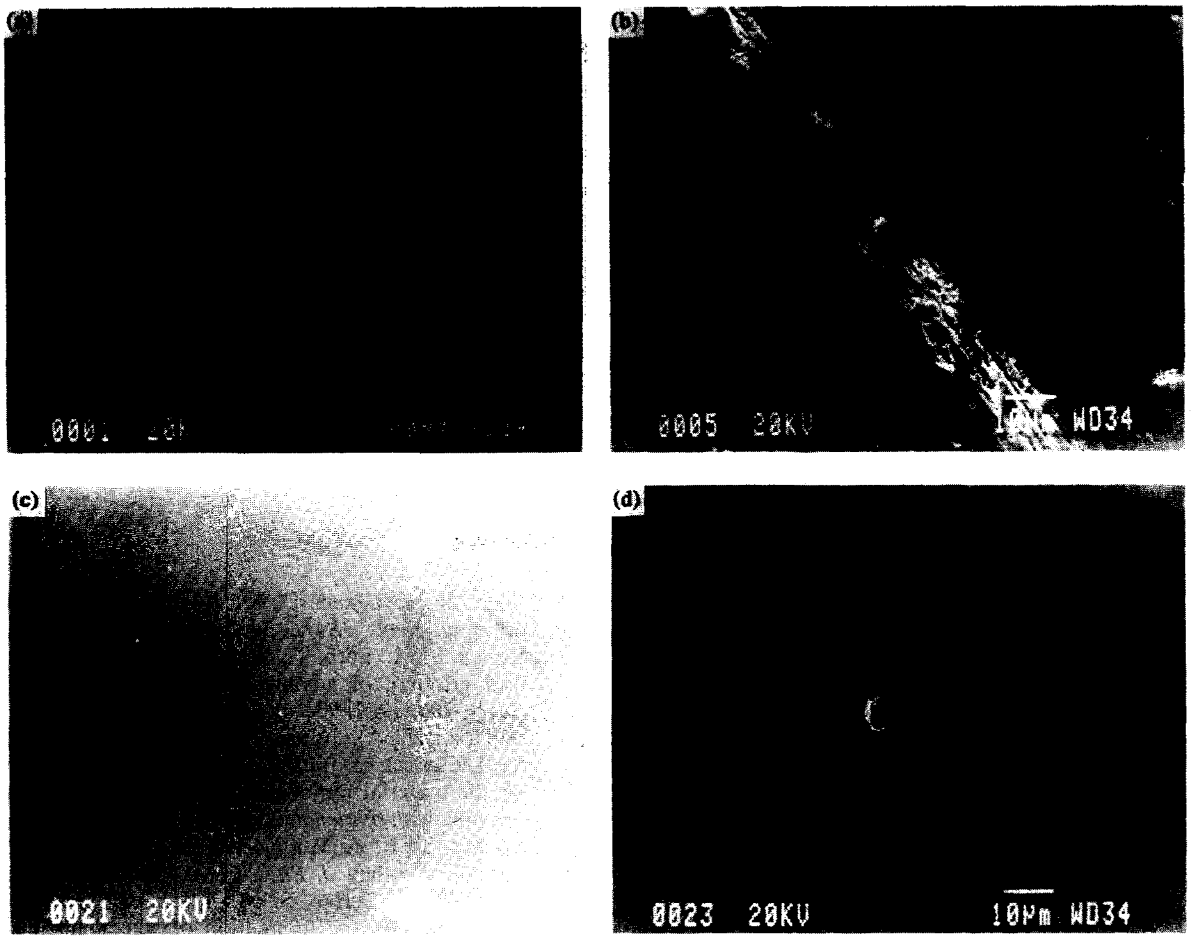

Fig. 15. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 1 wt% of 995 in 1 μm diamond slurry (a and b) and 1 wt% of 996 in 1 μm diamond slurry (c and d).

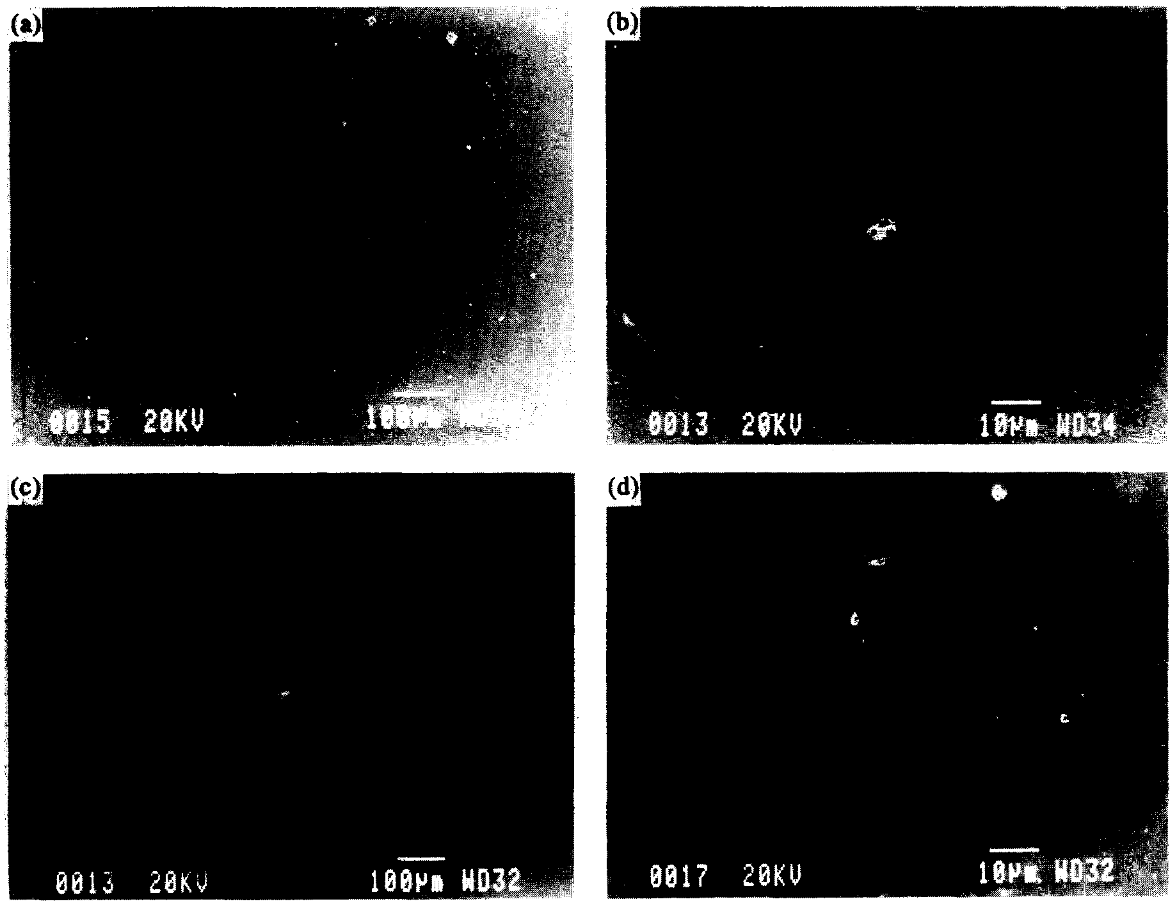

Fig. 16. SEM micrographs of ground surfaces after grinding tests of 1 h duration under 400 N load and 3000 rev min^{-1} speed in 1 wt% of 998 in 1 μm diamond slurry (a and b) and 1 wt% of NSE in 1 μm diamond slurry (c and d).

5 Conclusions

From the results reported in this paper, the following conclusions can be drawn.

(1) It has been found that the acid additive (A) and alkaline additive (B) are very effective additives to a diamond grinding slurry, increasing the rate of material removal from the silicon nitride balls. Inclusion of additive A or B into the mixture of 50% water-based and 50% oil-based diamond slurry increased the material removal rate six or four times, respectively.
(2) The material removal rate of the silicon nitride balls increases both with increasing concentration of inorganic additive (A or B) and increasing size of diamond particles in the slurry.
(3) Examination of ground surfaces revealed rough, pitted regions within a smooth surface. It was found that the surface produced by grinding in the diamond slurry with additive A or B is relatively rough.
(4) The presence of water in a diamond slurry increases the material removal rate, and the resulting surface is very smooth.
(5) The effect of three groups of organic additives on the rate of material removal was studied in the oil-based diamond slurry. A decrease in the material removal rate was noted in the presence of sulfur-containing additives. Addition of ester-based additives resulted in an increase of the material removal rate. The metal-containing sulfonate additives acted primarily as a viscosity improver. The significant increase in viscosity of the diamond slurry resulted in an increased material removal rate.

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