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A Study on Silicon Nitride Based Ceramic Cutting Tool Materials

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Abstract—The silicon nitride based ceramic cutting tool materials have been fabricated by gas pressure sintering (GPS) or hot pressing (HP). Their mechanical properties were measured and the effect of the fabrication variables on the properties were examined. Also, effect of adding TiN or TiC particulates on the mechanical properties of the silicon nitride ceramics were investigated. Ceramic cutting tools (ISO 120408) were made of the sintered bodies. Cutting performance test were performed on either conventional or NC lathe. The workpieces were grey cast iron, hardened alloy steel (AISI 4140, HRc>60) and Ni-based superalloy (Inconel 718). The results showed that fabrication variables, namely, sintering temperature and time, exerted a strong influence on the microstructure and mechanical properties of the sintered body, which, however, did not make much difference in wear resistance of the tools. High hardness of the tool containing TiC particulates exhibited good cutting performance. Extensive crater wear was observed on both monolithic and TiN-containing silicon nitride tools after cutting the hardened alloy steel. Inconel 718 was extremely difficult to cut by the current cutting tools.

Key words : Silicon nitride, TiC, TiN, Composite, Cutting tool, Sintering, Microstructure, Mechanical properties, Cutting performance.

1. Introduction

Advanced ceramic mateials are developed today to exhibit excellent hardness, oxidation resistance, wear resistance and other mechanical properties. The improvement of the mechanical properties of the sintered ceramic materials is able to partially satisfy the demand for a cutting tool for high speed machining aiming at higher productivity. Not only high speed machining but also cutting a certain hardened steel which had been machined by grinding is now feasible.

Silicon nitride is known to have well balanced mechanical properties among the ceramic materials. Silicon nitride is a material hard to sinter because of its low self diffusion coefficient resulting from the strong covalent bonding. Usually, sintering additives have been employed for promoting liquid phase sintering in order to obtain a product of high density. Microstructure of the sintered body varies depending on sintering variables as well as the chemical composition. Microstructure of the ceramic was reported to have a strong influence on the mechanical properties, especially on fracture toughness and strength [1]. Although there have been a lot of reports on the cutting performance of silicon nitride tools, it is hard to find a systematic research on the influence of the microsturcture and mechanical properties on the cutting performance or wear resistance of the silicon nitride [2].

Even though silicon nitride is known to have very good mechanical properties, it still needs to have higher fracture toughness and for some specific applications, it needs to be more non-reactive. Addition of TiN to silicon nitride was reported to increase fracture toughness of the composite over the monolithic silicon nitride unless particle size of TiN was not too big (d_{avg}. <10 micron). TiN particulates improved not only fracture toughness but also wear resistance possibly due to the formation of Ti-oxide surface layers [4]. Blanchard and Page also reported lower wear rates of Si_3N_4-TiC composites than those of Si_3N_4 or Si_3N_4-SiCw composite when tested against SiC ball [5]. They found that friction coefficients and wear rates of the Si_3N_4-TiC composite became lower as the test temperature increased while the specimens without TiC showed no change. They also referred to the Ti-oxide formed on the surface which lowered friction coefficient and wear rate at high temperature (>900°C). Besides, TiC or TiN is known to be less reactive with iron at high temperatures [6]. It needed studying if incorporation of TiC and TiN particulates in silicon nitride could provide better performance during cutting operation which often involved high temperature.

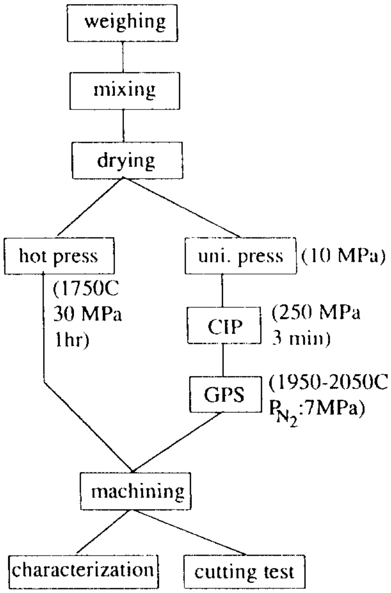


Fig. 1. Schematic flow diagram for preparation and characterization of the silicon nitride based ceramics.

Table 1. Ceramic powders used for this study

	Make	Grade	Avg. size (micron)	Others
Si_3N_4[1]	H.C. Stark	LC12SX	0.43	O:2.04%
Si_3N_4[2]	Ube	E10	0.55	O:1.19%
Y_2O_3	H.C. Stark	Fine	0.29	
Al_2C_3	Sumitomo	AKP30	0.37	
TiN	H.C. Stark	C.A.S	1.5	
TiC	H.C. Stark	B	1.5	

[1] Silicon nitride powder used for GPS specimens
[2] Silicon nitride powder used for HP specimens

Table 2. Nominal chemical compositions of the specimens (wt%)

	Si_3N_4	Y_2O_3	Al_2O_3	TiN	TiC
GA[1]	93	6	1		
GB[1]	93	5	2		
HA[2]	93	5	2		
HB[2]	48	4	1.5	46.5	
HC[2]	70.7	4.5	1.8		23

[1] Sintered by GPS
[2] Sintered by HP

2. Experimental Procedures

Specimens were prepared according to Fig. 1 with ceramic powders shown in Table 1. Table 2 shows nominal chemical compositions of the powders. Two different kinds of silicon nitride powders were used for this study. One was the product of H.C. Stark Co., Germany. This powder was fabricated by direct nitridation of metallic silicon. The other was the product of Ube Co., Japan. This was fabricated by decomposition of di-imide process and was known to have narrower particle size distribution.

Powders of the appropriate compositions were mixed by ball milling for 72 hrs after weighing. Drying of the mixed slurry was carried out on hot plate to evaporate ethanol while keeping the magnetic spin rotating and preventing segregation of the powders. The prepared dry powder mixture was either hot pressed or sintered by gas pressure sintering. For GPS, the green compact was formed by uniaxial press under 10 MPa followed by Cold Isostatic Press (CIP) under 250 MPa prior to sintering. After sintering, dense specimens were cut and ground. Specimens for mechanical property measurements and microstructural observation were finished by polishing with 1 micron diamond paste. Ceramic cutting tools were machined to have ISO 120408 shape. Cutting tests were carried out on either conventional type lathe or NC lathe (Hwa-Cheon Co., Korea, Ecostar 3). Workpiece materials used were 1) grey cast iron (d=110 mm, l=300 mm), 2) hardened alloy steel (AISI 4140, HRc 60, d=60 mm, l=250 mm), and 3) Inconel 718 (d=126 mm).

3. Results and Discussion

3-1. Gas Press Sintered Si_3N_4

Two specimens GA and GB were sintered by GPS. They were sintered either at 1950°C for 2.5 hrs (schedule I) or at 2050°C for 4hrs(schedule II) in nitrogen atmosphere (P_{N2}: 7 MPa). Table 3 shows the properties obtained including density, microvickers hardness, fracture toughness, 3 point flex-

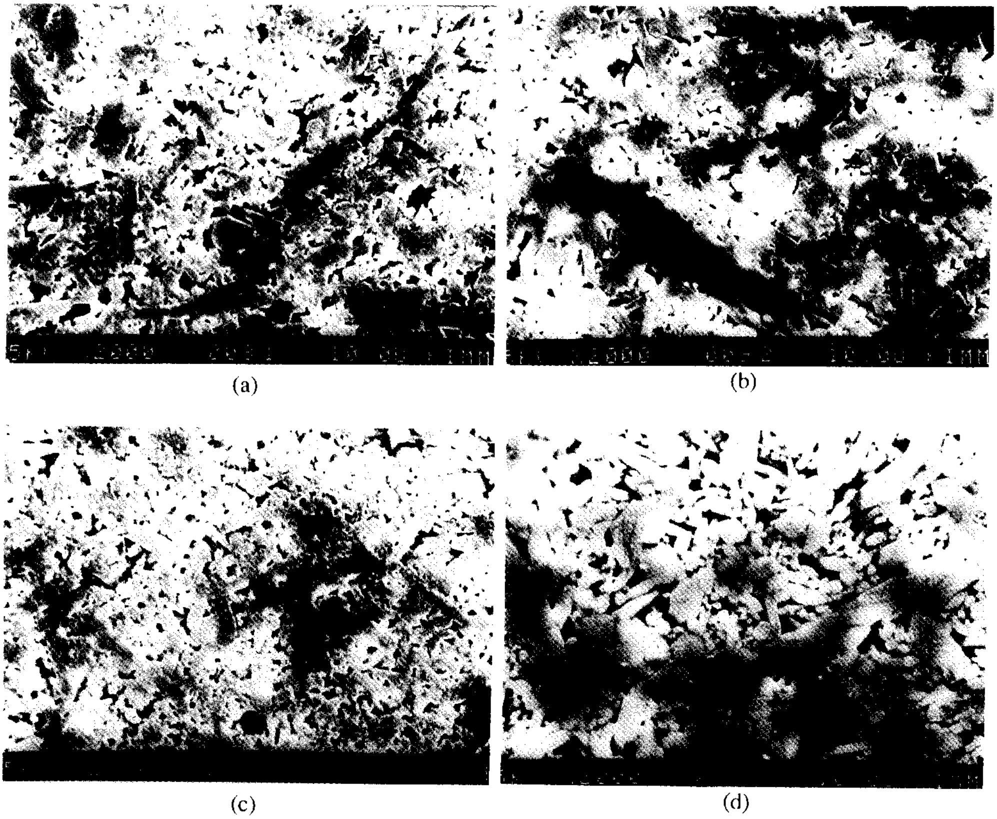
Table 3. Properties obtained for the specimens GA and GB (numbers in () are deviations)

property \ spec GPS	GA I	GA II	GB I	GB II
density (%)	99.5	99.6	99.2	99.5
hardness, Hv1kg (kg/mm^2)	1540 (34)	1486 (34)	1535 (40)	1514 (35)
fracture toughness (MPa*m$^{1/2}$)^1	6.43 (0.4)	7.01 (0.43)	6.5 (0.42)	7.19 (0.35)
3 point flexural strength (MPa)^2	960 (48)	857 (44)	920 (50)	868 (15)
" at 1000°C	453 (60)			473 (50)

[1] Indentation crack length method, 10 kg load, Evans and Charles' equation [7].

[2] Statistics based on 6-9 measurements, each.

ural strength and others. The results show that the specimen were fully densified although GB seemed a little bit harder to densify than GA. Microvickers hardness values were close to one another and were in the range between 1450 and 1550 kg/mm^2. The specimens sintered at higher temperature (schedule II) exhibited lower hardness in spite of higher density obtained. Higher fracture toughness values were obtained for the specimens sintered according to schedule II than those according to schedule I. Also, specimen GB showed slightly higher K_{IC} than GA. The flexural strength measured at RT was higher for the specimens sintered at lower temperature (schedule I). Flexural strength measured at 1000°C showed a significant decrease from the RT measurement. In summary, as the sintering temperature increased, density and fracture toughness increased while hardness and flexural strength decreased. Mechanical properties obtained were closely related



(a) (b) (c) (d)

Fig. 2. SEM micrographs of GPS'ed monolithic silicon nitride specimens; (a) GA sintered according to schedule I, (b) GA according to schedule II, (c) GB according to schedule I and (d) GB according to schedule II.

to the microstructures which were significantly influenced by the sintering schedule. Fig. 2 shows the microstructures of the specimens. Coarser microstrucutres were obtained at higher sintering temperature. Fracture toughness of silicon nitride ceramics which contained the whiskerlike elongated grains increased as the size of those grains increased. Also, coarser microstructures often resulted in lower strength and lower hardness [1,2], even though the latter was influenced to much less degree than the former. However, high temperature strength was almost the same as for both GA (schedule I) and GB (schedule II) specimens in spite of the difference in the microstructures resulting from different sintering schedules. Also, it was noted that strength measured at 1000°C was 400-500 MPa lower than that at RT. There had been a rapid decrease in strength between RT and 1000°C. This can be explained by the fact that all the grains were surrounded by the glass phase which exhibited rapid softening at high temperature. Softening of the glass according to increase of temperature is critically dependent on its chemical composition. GA and GB had similiar chemical compositions to each other, which was resposible for the similiar high temperature strength values obtained.

3-2. Hot Pressed Si_3N_4 and its composites

Three specimens HA, HB and HC were made by hot pressing at 1750°C for 1 hr under 30 MPa. Table 5 shows the properties measured for the specimens (HA, HB and HC). All the specimens were fully dense after hot pressing. It is interesting to see that higher sintering density was obtained for the composites than for the monolithic silicon nitride. Usually, the composites were harder to densify than the monolithic one due to the foreign particles dispersed in the matrix inhibiting mass transport. However, TiN was reported not to inhibit densification [8] and to promote alpha to beta phase transformation of a silicon nitride ceramic [9]. TiC could cause troubles during sintering by reacting with Si_3N_4 and generating gas upon extended sintering schedule [10]. Specimens HC was apparently sintered successfully to the full density before the problem seriously inhibited densification. Microvickers hardness value of HA was very similair to that of GB in spite of the difference in the Si_3N_4 powders used and the grain sizes of the sintered bodies. HB exhibited a similiar hardness value as HA, while HC had much higher hardness than HA. TiC is well known to have very high hardness ($Hv,\ _{100\ g}$=3200 kg/mm^2)^{11} and this was well expressed by high hardness of HC. TiN has much lower hardness ($Hv,\ _{50\ g}$=1800 kg/mm^2) than TiC and was reported to have a similiar hardness to that of silicon nitride [11]. So, HB containing TiN showed a similiar hardness to that of the monolithic HA. Fracture toughness of HA was almost the same as that of GB notwithstanding the difference in microstructures. Hot pressing aligned the elongated grains in the specimen normal to the hot pressing direction while gas pressure sintered specimen had the grains randomly oriented. Hot pressed specimen was reported to exhibit higher fracture toughness on the plane normal to the hot pressing direction than on the plane parallel to it [12]. The aligned microstructure of HA gave fracture toughness as high as GB. TiN was contributed to the improvement of K_{IC} of HB, as expected. However, HC which contained TiC exhibited lower fracture toughness than the monolithic HA. This was quite surprising because higher thermal expansion coefficient of TiN than the silicon nitride matrix in HB caused thermal residual stress and microcracking around TiN particles and improved fracture toughness, and TiC particles which also had higher thermal expansion coefficient than the silicon nitride were expected to be able to improve fracture toughness, too. Blanchard and Page reported higher fracture toughness for the Si_3N_4-TiC composite than the monolithic Si_3N_4. It is not clear at this point why HC exhibited such a low fracture toughness. HA showed high flexural strength, 1284 +/- 117 MPa, expecially if compared with GB of the same nominal chemical composion. HA had much finer grains

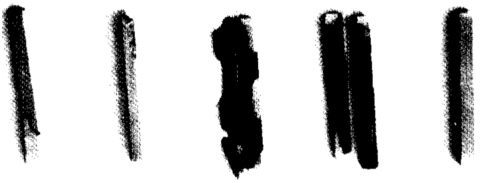
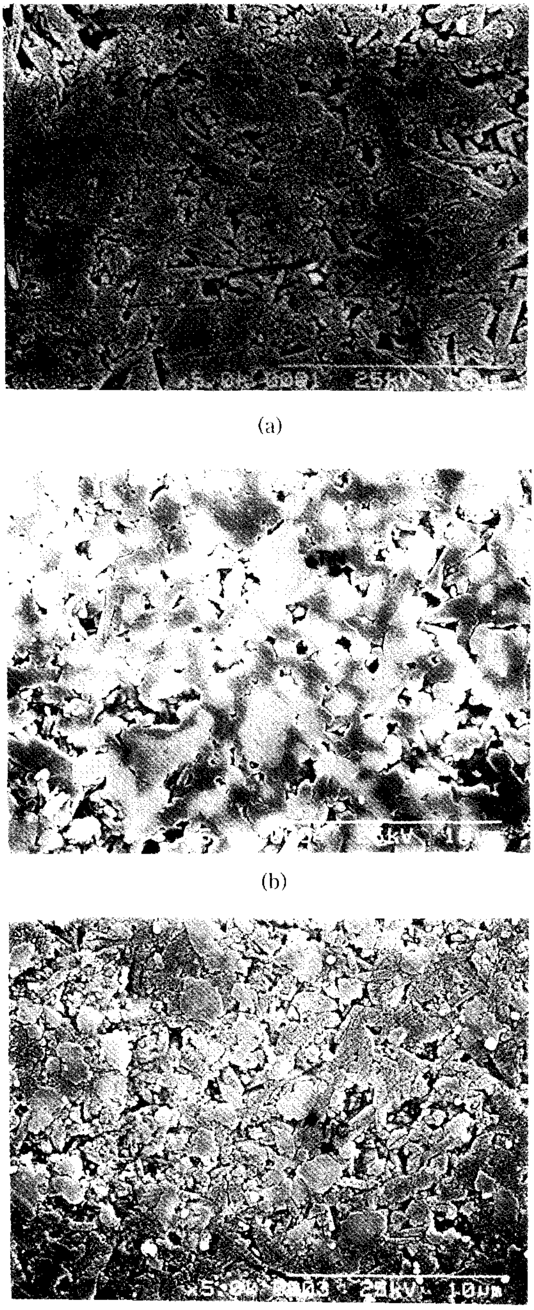


Fig. 3. Picture of HC specimens cracked by thermal shock; specimen was water quenched from 900°C.



(a)

(b)

(c)

Fig. 4. SEM micrographs of (a) HA, (b) HB and (c) HC; foreign particles, TiN or TiC, inhibited grain growth of the matrix silicon nitride grains.

than GB (Fig. 2 and Fig. 4) and the fine accicular grains were aligned normal to the crack propagation direction (analogy to plywood). One more possible reason for higher strength of HA over GB was the difference in the Si_3N_4 powders used. Si_3N_4 powder used for HA was more refined and had narrower particle size distribution than that for GB. More uniform particle size distribution of the starting powder would make finer and more uniform grain size in sintered body than wider particle size distribution because a grain in silicon nitride was reported to keep growing until it hit another grain of a same diameter during sintering. So, large grains would grow and fine grains would disappear near the large grain or would remain small away from it in case of wide particle size distribution. But a grain would grow and soon, it would stop growing upon hitting the other grain of a same size in case of narrow particle size distribution. High temperature strength of HA was much higher than GB. The difference in the high temperature strength values of HA and GB pointed out that the chemical nature of the starting powder exercised a strong influence upon them. HB showed 1050 +/- 53 MPa of the 3 point flexural strength at RT. HC showed low strength 746 MPa and big variability (+/-242 MPa). Also, HC was very vulnerable to thermal shock as shown in Fig. 3 which was taken after water quenching from 900°C. The specimen was shattered, which was not observed for HA or HB after the quenching. So, low fracture toughness and big variability in strength measurement of HC were thought to be very closely related to its vulnerability to thermal shock which might be applied during cooling from the sintering temperature.

Fig. 4 shows the microstructures of HA, HB and HC. HB and HC showed smaller grain size of matrix silicon nitride than that of HA, which suggests that TiN or TiC particulates inhibited growth of the silicon nitride grains. Part of TiN and TiC particulates in HB and HC, respectively, fell off the specimen upon etching.

3-3. Cutting Tests

Five kinds of sintered silicon nitride based ceramics, GA, GB, HA, HB and HC were cut and ground to have ISO specification SNGN120408 with -20° × 0.2 mm edge chamfer. Grey cast iron was cut on the conventional lathe by using the tools made of GA and GB sintered according to schedule I and II, respectively. Cutting speed, feed and depth of cut

Table 6. Flank wear measured after cutting grey cast iron

tool	cutting time	Flank wear (mm)	lathe
GA	11'20"	0.38	Conv.
GB	11'45"	0.39	Conv.
GB	4'59"	0.18	N.C.
HA	5'01"	0.21	N.C.
HB	4'25"	0.24	N.C.
HC	6'19"	0.22	N.C.

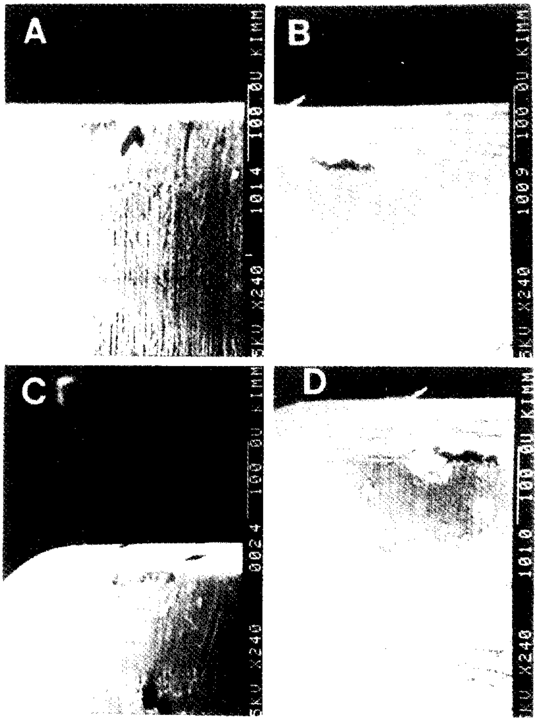


Fig. 5. SEM micrographs of the cutting edge of the tool worn during cutting grey cast iron; (a) GA, (b) HA, (c) HB and (d) HC tool tested on NC lathe, cutting speed: 450 m/min, feed: 0.1 mm/rev., depth of cut: 1.5 mm.

Table 7. Test results for resistance to chipping or breakage (workpiece: AISI 4140)

feed (mm/rev)	speed(m/min)	120	150	180
0.1	HB		O	X
	HC	O	O	O
0.12	HB	O	X	
	HC		O	△
0.15	HC		O	△

O: no chipping or breakage
△: chipping but still cutting
X: breakage

were 1200 rpm, 1.7 mm/rev., and 1.5 mm respectively. Table 6 shows the test results of cutting grey cast iron. GA showed 0.38 mm flank wear after 11 min's and 20 sec's cutting, while GB exhibited 0.39 mm after 11 min's and 45 sec's. There was no noticeable difference between the tools made of GA and GB in spite of the significant difference in the material properties. The tools made of GB, HA, HB and HC were tested to cut grey cast iron on the NC lathe. Test was carried out at V=450 m/min, feed=0.1 mm/rev, and depth of cut=1.5mm. HA tool showed 0.21 mm flank wear after 5'01" while GB showed only 0.18 mm after cutting for a comparable time. Higher fracture toughness, hardness and density of GB were the possible explanation for lower flank wear of GB tool than that of HA tool. Also, tensile stress was applied at a certain angle to the plane of accicular grains, which might lower wear resistance of the tool. HB tool containing TiN exhibited the lowest wear resistance among the hot pressed tools in spite of the highest fracture toughness, while HC of highest hardness showed very good tool wear resistance. Therefore, hardness of the tool played an important role in cutting grey cast iron while fracture toughness resulting from microcracking as shown in HB tool might not improve wear resistance of the tool. Also, it is worth attention that flexural strength of tool material was hard to be related to performance of the tool. Fig. 5 shows the SEM micrographs of cutting edge after cutting grey cast iron.

Heat treated alloy steel (AISI4140, HRc 60) was used for cutting performance test of HB and HC tools. GB tool was initially tested, but it exhibited an extensive crater wear on rake face. So, monolithic silicon nitride tools including GA, GB and HA were not tested. Silicon nitride is known to dissolve in steel at high temperature. First set of cutting tests were performed on NC lathe to examine resistance to chipping or breakage of the tool. Table 7 shows the results obtained when depth of cut was fixed at 0.5 mm and speed and feed were varied. Both HB and HC tools exhibited either chipping or breakage under more severe cutting conditions than the crit-

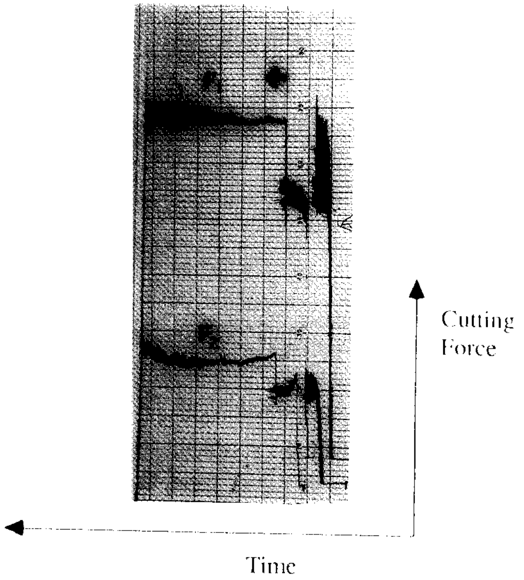


Fig. 6. Variation of the cutting forces measured during cutting the hardened alloy steel by HC tool; F1: principal force, F2: normal force.

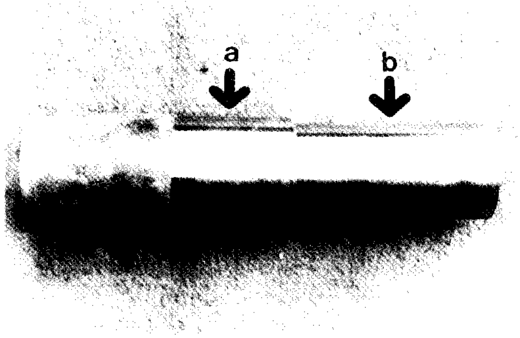


Fig. 7. Picture of the workpiece after cutting test; surface cut by (a) the alumina-TiC tool looks smoother than that by (b) HC tool.

Fig. 8. SEM micrographs of the cutting edge of the tool worn during cutting Inconel 718; (a) GA, (b) HA, (c) HB and (d) HC tool tested on NC lathe, cutting speed: 450 m/min, feed: 0.1 mm/rev., depth of cut: 1.5 mm.

ical ones. HB containing 35 v/o of TiN showed a serious crater wear and lower resistance to breakage than HC containing 20 v/o TiC. TiN was reported to be more chemically stable against iron at high temperature than TiC, not to mention Si_3N_4[6]. From chemical stability point of view, HB should have shown better performance than HC. Therefore, not only chemical stability but also mechanical property, especially hardness in this case, should be counted for resistance to chipping or breakage of the tool. For comparison, a commercial alumina-TiC cutting tool was tested under the same conditions as shown in Table 7. The alumina-TiC tool exhibited a similiar results as HB tool. So, as far as resistance to breakage was concerned, inspite of the chemical unstability against steel at high temperature, HC, Si_3N_4-TiC, tool performed very well, even better than alumuna-TiC tool which ususally was recommended for this kind of cutting operation. Flank wear of the tool was measured after cutting 8 passes (for 28' 15") at speed of 120 m/min, feed 0.1 mm/rev, and depth of cut 0.5 mm. The results showed that HC tool exhibited flank wear of 0.32 mm and the alumina-TiC tool exhibited 0.28 mm. Fig. 6 showed the cutting force measured during test with HC tool cutting AISI4140. F1 and F2 represents principal and normal force, respectively. Average cutting force slightly increased, but the force vibrated around the average value and the amplitude became increased significantly as cutting proceeded. Machined surface of the workpiece was rougher for HC tool than for alumina-TiC tool (Fig. 7). HB tool was broken in 8

Table 8. Flank wear (mm) measurements during cutting Inconel 718; V=20 m/min, feed=0.15 mm/rev, DOC=1 mm

time (min) tool	0.5	1	2	4
GB	NM	0.73	1.28	1.386
HA	NM	0.498	0.599	0.795
HB	0.693	0.833	1.063	1.923
HC	0.561	0.647	0.688	1.053

passes.

In case of Inconel 718, all of the tools tested did not perform to the satisfaction. All the tips were heated to yellow hot to the naked eye during cutting even with a coolant applied. Inconel 718 is known to have low thermal conductivity (11.2 w/mk) compared with that of a low carbon steel (51.9 w/mk[13]). Lower thermal conductivity of the workpiece drove all the heat generated during cutting to rake surface of the tool and heated it to very high temperature. Table 8 shows the results of flank wear measurements during cutting. HA of the highest strength and HC of the highest hardness exhibited better cutting performance. However, all the tools showed extensive DOC notch and wear of the tool was too much to be practically meaningful as a tool. Fig. 8 shows the damaged cutting edge after the test with Inconel 718.

4. Conclusions

The silicon nitride based ceramic cutting tools were fabricated and evaluated.

Following conclusions were obtained.

(1) Sintering temperature and time exerted a strong influence upon the microstructure and mechanical properties of the monolithic silicon nitride; higher temperature and longer holding time produced coarse microstructure with lower strength and higher fracture toughness.

(2) Incorporation of TiN and TiC particulates imporved fracture toughness and hardness of the composite, respectively.

(3) In case of cutting grey cast iron, the two GPS'ed monolithic silicon nitride tools exhibited no difference in cutting performance in spite of the difference in the microstructures and mechanical properties, and the composite containing TiC showed good tool wear resistance.

(4) In case of hardened alloy steel, the tool made of the composite containing TiC cut better than that containing TiN or the monolithic silicon nitride tool.

(5) Inconel 718 with very low thermal conductivity was hard to cut satisfactorily. Tetative conclusion on cutting performance tests is that the monolithic silicon nitride of fine microstructure performed best among the tools tested.

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