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SiAlON based ceramic cutting tools

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Abstract

There is a continuous need for improving the existing cutting tool materials, which is driven by a strong competition on the market and also by more difficult to machine materials like, e.g. high alloyed grey cast iron. Ceramic cutting tools offer a high productivity due to their excellent hot hardness, which allows high cutting speeds. Under such conditions the cutting tool must be resistant to a combination of mechanical, thermal and chemical attacks.

The paper shows the potential of new alpha-beta-SiAlON cutting tools reinforced with SiC, WC, $MoSi_2$ or Ti(C,N) particles. The microstructure, mechanical properties and wear resistance of these composites will be presented.

Keywords: Cutting tools; SiAlON; Hardness; Wear resistance

1. Introduction

One group of the broad range of ceramic products are ceramic cutting tools, which allow the machining of metal parts at very high speeds. These cutting tools can be divided into a few main material classes: silicon nitride, zirconia toughened alumina and mixed-ceramics, which are composites of zirconia toughened alumina and titanium carbonitride, and cubic boron nitride (Table 1). Especially silicon nitride based materials are excellent for machining cast iron because of their combination of high fracture toughness, hot hardness and thermal shock resistance. Roughly two-third of CeramTec's insert production are silicon nitride based materials. Recently, alpha-beta-SiAlONs gained attention as cutting tool materials due to their superior hardness over silicon nitride. Nevertheless, in certain applications alumina-based cutting tools are still the material of first choice because of their high chemical stability.

For several years now cutting tools based on alpha-beta-SiAlONs are in service. Adding hard particles like for example silicon carbide to the SiAlON matrix is a possible way to further improve the wear resistance and increase life time. Different research groups have investigated the properties of particle-reinforced silicon nitride and SiAlONs. These results are summarized in some reviews.[1,2] Even cutting tools made of Si_3N_4–TiN-composites had been on the market more than 15 years ago, but disappeared in the meantime—probably because of high manufacturing costs. SiAlONs, however, possess a better sinterability than silicon nitride and offer more possibilities in the development of particulate composites, so that such composites have gained new attention recently. Thus, particle-reinforced SiAlONs are promising candidates for further improving the wear resistance of ceramic cutting tools. One aim is to increase the hardness by the addition of hard particles like e.g. silicon carbide and at the same time to maintain the good fracture toughness of the matrix for the composite material. SiAlON–SiC-composites have already been established successfully in the cutting tool market by CeramTec. In this work a first attempt was made to use other particles than SiC like WC, $MoSi_2$ and Ti(C,N) for SiAlON-composites for cutting tool applications. Although these composites did not produce satisfying results, they showed potential for further optimization. The properties and application test results of these composite materials will be discussed.

2. Experimental procedure for producing SiAlON-composites

Various SiAlON-composites with particle additions up to 30 or 35 vol% were made by adding the powder to the raw materials of a Mg-Y-SiAlON-composition with a nominal alpha/beta-SiAlON-phase ratio of 20:80. Four different reinforcement

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Table 1
Overview on typical properties and applications of cutting tools [CeramTec]

Cutting material	Oxide-ceramic	Mixed-ceramic	Silicon nitride ceramic	Cubic boron nitride (cBN)
Colour	White	Black	Grey or green (SiC-composite); gold or maroon when coated	Black; gold when coated
Strength, toughness, hardness	600 MPa, 6 MPa m$^{1/2}$, 1650 HV10	750 MPa, 5 MPa m$^{1/2}$, 1900 HV10	1000 MPa, 5–6 MPa m$^{1/2}$, 1600–1700 HV10	700–1500 MPa, 4–7 MPa m$^{1/2}$, 2500 HV10
Main features	Finish turning of cast iron.	Hardened steel; super finishing	Withstands heavy interrupted cuts	Excellent surface finishing; hard machining;

particles, which are commercially available products, were used: α-SiC powder (D_{50} = 0.8 μm), Ti($C_{30}N_{70}$) (D_{50} = 0.8 μm), WC (D_{50} = 1.5 μm) and $MoSi_2$ (D_{50} = 2.5 μm). With increasing SiC content the amount of sintering additives, which could not be incorporated into the Mg-Y-α-SiAlON, was linearly increased, so that the SiAlON–30 vol%SiC-composite, for example, contains an surplus of sintering additives of approximately 3.3 vol% compared to the matrix itself. Some of the excessive sintering additives are necessary to wet the surface of the added particles.

After milling and spray-drying the powder was consolidated by uniaxial pressing. Binder burnout was carried out in nitrogen at 550 °C. Sintering was done by two-step gas pressure sintering at 1930 °C and 20 bar nitrogen and 80 bar argon pressure for 3 h. All samples were sintered with the same time–temperature-profile. The microstructure was characterized by SEM after plasma etching. From Vickers indentations the hardness (HV10) and fracture toughness (using Palmqvist model)[3] have been analysed. The phase content was measured using X-ray diffraction. The alpha/beta-SiAlON ratio was determined after Gazzara and Messier[4].

The wear resistance was evaluated in a turning test by roughing a grey cast iron (GG15, v_c = 1000 m/min, f = 0.50 mm/rev, a_p = 2.0 mm). The length of the wear mark was measured during testing in intervals of 32 cuts.

3. Results and discussions

3.1. SiAlON–SiC-composites

Silicon carbide is known for its high hardness and also for its compatibility with silicon nitride during sintering. Final densities of >99% of theoretical density could be achieved up to a SiC content of 30 vol%.[5] The SiC particles do not participate in liquid phase sintering under the used conditions and therefore hinder sterically the densification of the SiAlON matrix. Yet, even with 30 vol% SiC full density could be achieved. However, the sinterability of samples with 35 vol% SiC is too low. These samples obtained a density of only 97% under the used sintering temperatures.

The microstructure of the SiAlON-matrix consists of acicular beta' grains, globular alpha' grains and a grain boundary phase. The SiC particles are located at the grain boundaries and also inside the beta-SiAlON grains (Fig. 1). It seems that the smaller SiC particles are preferably incorporated into the growing SiAlON grains during sintering, whereas the bigger ones are left at the grain boundaries. Higher amounts of SiC lead to a finer microstructure with less acicular beta' grains. This is because the more SiC particles are present the more they hinder the grain growth of the beta' grains.

As expected the *hardness* of the alpha/beta-SiAlON can be increased by the addition of SiC (Fig. 2). With 25 vol% SiC an increase in hardness of approximately 25% can be achieved. The hardness does not linearly increase with the SiC content. Above 25 vol% SiC hardly any improvement of hardness can be observed. This is due to the fact that with higher SiC addition more sintering additives had to be added to the composition. More sintering additives cause more glassy phase after sinter-

Table 2
Density, mechanical properties and SiAlON-phase content of sintered Ti(C,N)-, WC- and $MoSi_2$-SiAlON-composites

Particle addition (vol%)	Density ($g\,cm^{-3}$)	HV10 (GPa)	K_{IC} ($MPa\,m^{1/2}$)	α/β-SiAlON-ratio
None	3.250	16.0	7.0	20:80
10% Ti(C,N)	3.457	17.3	6.3	33:77
20% Ti(C,N)	3.675	17.7	6.1	45:55
30% Ti(C,N)	3.893	18.4	6.1	66:44
10% WC	4.518	18.5	6.1	56:54
20% WC	5.785	18.8	5.9	100:0
30% WC	7.252	17.2	5.8	100:0
10% $MoSi_2$	3.601	14.8	5.5	12:88
20% $MoSi_2$	3.750	14.8	5.9	8:92
30% $MoSi_2$	3.972	14.5	6.3	0:100

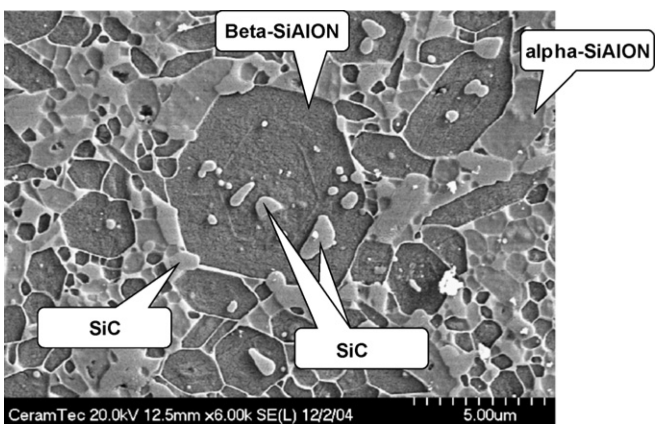

Fig. 1. Microstructure of SiC-composites with 10 vol% SiC (SEM, plasma-etched).

ing which lower the materials hardness. The steep decrease in grain size with addition of small quantities of particles may also contribute to this.

Fracture toughness was calculated by measuring the crack lengths caused by the hardness indentations. Up to 15 vol% of added SiC the toughness remained almost constant (Fig. 2). Above that a significant decrease in toughness was observed. Most probably the finer microstructure of the higher SiC containing composites caused the decrease in toughness.

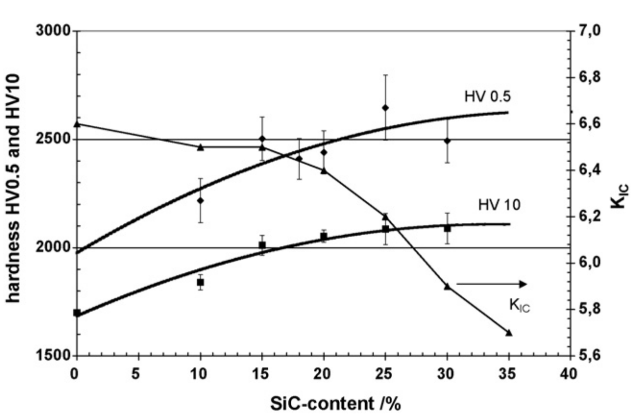

Fig. 2. Vickers hardness (HV0.5 and HV10) and indentation fracture toughness of SiC–SiAlON-composites.

3.2. Other SiAlON-composites

SiC is said to be thermodynamically not stable in contact with iron-containing metals. This means that during a machining operation a chemical wear occurs because of reactions between SiC and the iron of the metal. However, the same is true for silicon nitride or SiAlONs itself but in a less extend, so that the machining of steel, for example, is not possible due to high temperatures and therefore high chemical wear. In any case, instead of SiC alternative particles for SiAlON-composites would be more favourable. Of course, any added reinforcement particle should be stable during sintering of the SiAlON-composite. Thermodynamic considerations can help in sorting suitable compounds. It is known that $Ti(C_{30}N_{70})$ can be sintered together with silicon nitride without degradations or unwished reactions.[6] Tungsten carbide, on the opposite, is said to be unstable, forming SiC and W or tungsten silicides depending on the nitrogen content. However, previous screening tests showed that WC-composites can indeed be achieved under the used conditions. Also there is some work published on tungsten carbide–silicon nitride.[7] $MoSi_2$ often changes to Mo_5Si_3, a less Si-rich phase. This reaction is strongly depending on the nitrogen pressure.[8]

Whereas Ti(C,N) and WC can increase the hardness of an SiAlON-composite, the addition of $MoSi_2$ was found to affect the fracture toughness positively.[9,10]

These three types of SiAlON-composites were prepared in the same way like the SiC-composite. All samples were sintered under the same conditions and were easy to densify; even at 30 vol% particle addition near full densification was achieved (Table 2). When sintering silicon nitride usually a small mass loss is observed. In the case of $MoSi_2$ a mass increase of 5.4% for the sample with 30 vol% added $MoSi_2$ was measured. As mentioned before, $MoSi_2$ is not stable and converts mostly to Mo_5Si_3 and Si_3N_4 by the reaction with nitrogen, which explains the mass gain. An optimization of the nitrogen pressure could prevent this reaction.[8]

From the experience with the SiC-composites it was expected that the phase composition after sintering would consist of approximately 20% alpha- and 80% beta-SiAlON, apart from further crystalline phases in the grain boundary like mellilite and, of course, phases from the particle addition. This could be confirmed for the Ti(C,N) containing material. However,

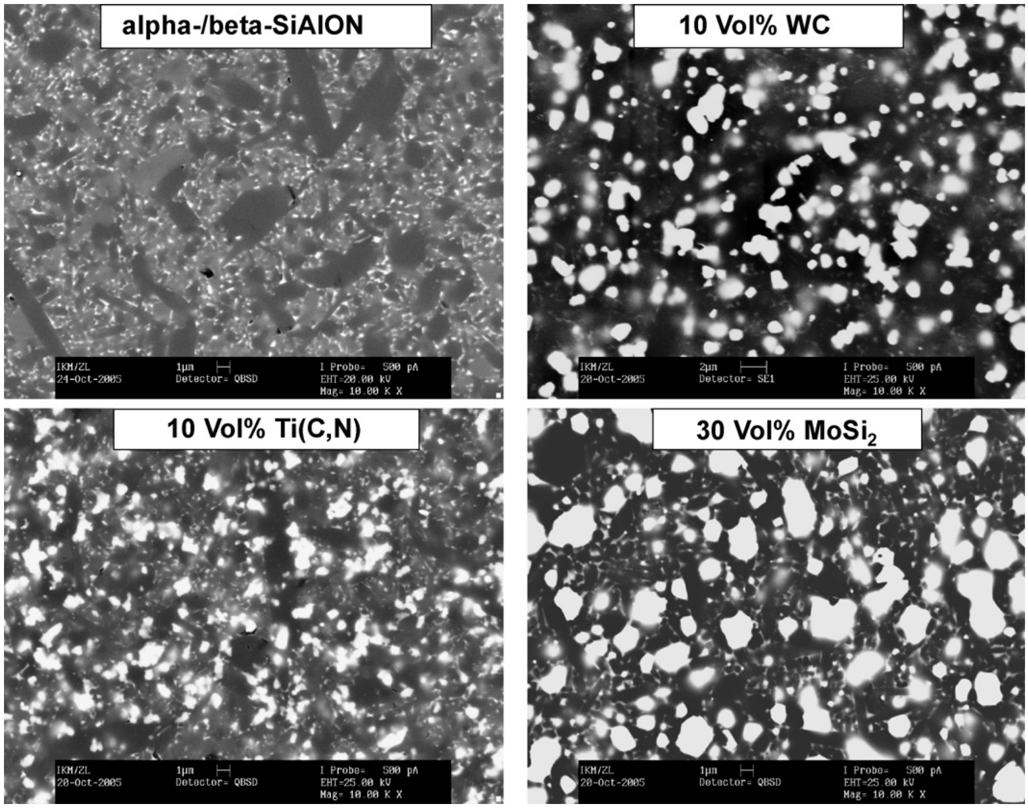

Fig. 3. Microstructure of composites with 10 vol% Ti(C,N) and WC or 30 vol% $MoSi_2$-addition in comparison with alpha/beta-SiAlON matrix (SEM).

with more Ti(C,N) present the higher was the alpha' content, caused most probable from the mentioned surplus of sintering additives. In the samples with prior $MoSi_2$-addition strong intensities of Mo_5Si_3 and only weak $MoSi_2$-peaks were found by X-ray diffraction. Furthermore the $MoSi_2$-addition lowered slightly the alpha/beta-phase ratio and promoted the crystallization of Y-silicates. Whether the added WC in the sintered sample was still present as the carbide or reacted to other compounds was not clear in the beginning. Silicides were clearly not detected by XRD but it is difficult to distinguish between tungsten carbide and nitride. By EDX analysis in the SEM it was found out that the tungsten containing particles showed significant less nitrogen and more carbide than the SiAlON matrix. Thus, it was concluded that WC is indeed present after sintering. Most unexpected, the WC-addition influenced strongly the SiAlON-phase ratio. When 20 vol% WC were added no beta-SiAlON-peaks could be detected anymore! To explain this behaviour a more detailed investigation would be necessary.

The microstructure of these composites are similar to the SiAlON–SiC-composite (Fig. 3), but particles inside the SiAlON-grains could not be observed. All added particles are located at the grain boundaries. The size of the particles was found to be independent to its volume ratio in the composite, indicating that no significant agglomeration during sintering took place. Only for the highest volume ratio of WC, i.e. 30 vol%, some agglomerated particles could be found. The WC powder had the broadest grain size distribution and the largest grains whereas the Ti(C,N) powder was quite fine. The above-described changes in the alpha/beta-phase ratio can be seen in the microstructure, as well.

By the addition of Ti(C,N) the hardness increased from approximately 1600 up to almost 1900 HV10 (Fig. 4). The toughness decreased at the same time from approximately 7 to 6 MPa $m^{1/2}$ (Fig. 5), which would still be sufficient for most of the machining applications. The samples with $MoSi_2$-addition showed a different behaviour. Here, the hardness decreased continuously with higher $MoSi_2$-addition. This can be explained by the parallel decrease in alpha-SiAlON content. The reduction of the alpha-phase, which makes a substantial contribution to the hardness of the material, cannot be compensated by the resulting molybdenum silicides. Unfortunately, there was even no positive

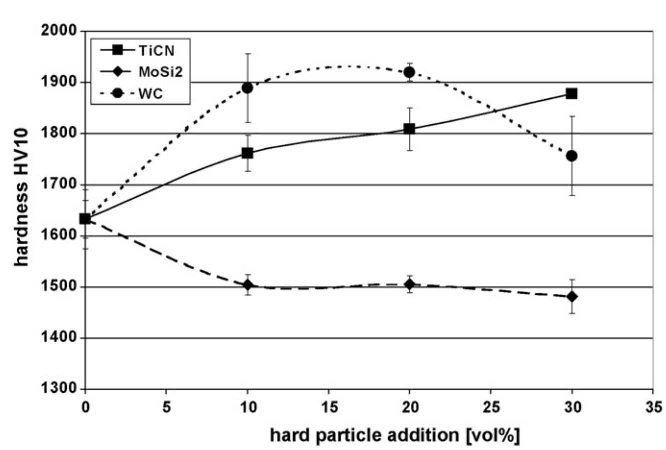

Fig. 4. Hardness of various SiAlON-composites (HV10).

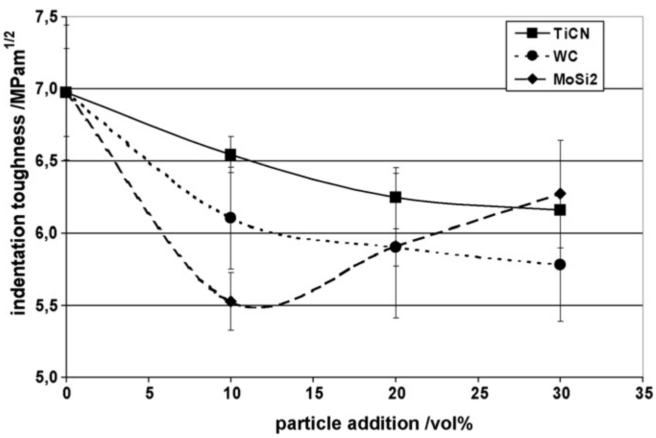

Fig. 5. Indentation toughness of various SiAlON-composites.

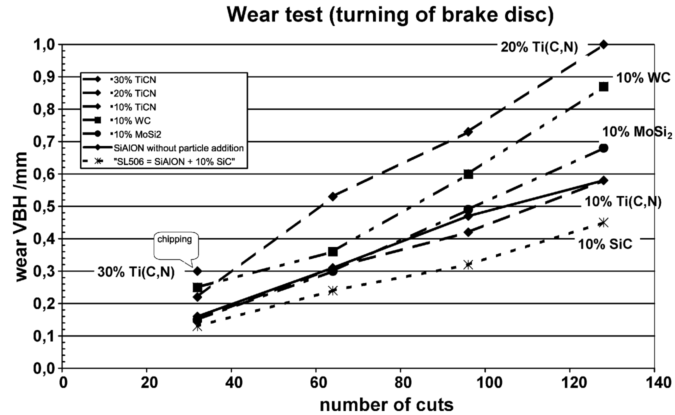

Fig. 6. Machining test results of different SiAlON-composites.

effect on toughness. Instead, slightly lower toughness values as for the matrix itself were measured (Fig. 5). The WC-composites showed a strong increase in hardness, which is not only caused by the WC itself but by the increase in the alpha/beta-phase ratio. When the WC content was increased to 30 vol% WC the hardness dropped down from approximately 1900 to less than 1800 HV10. This was very unexpected because the composites with high WC content consist of nearly 100% alpha-phase in the matrix and therefore should exhibit a very high hardness. By optical microscopy some porosity could be observed but because of their faceted shape it is supposed that these were introduced during grinding and polishing. Maybe internal stresses due to the large thermal expansion mismatch of the WC grains and the matrix lead to such behaviour. The toughness of all WC-samples was quite low.

3.3. Evaluation in machining tests

The measurement of hardness and toughness give static material properties but only limited hints if a material is suitable as a cutting tool. Much more important is the performance in real *machining tests*. Roughing of brake discs made of cast iron is a typical application for silicon nitride based cutting tools. In this operation the hard and irregular shaped casting skin has to be removed. Because of environmental and economical reasons this process is carried out without a coolant. Thus, temperatures at the cutting edge can exceed 1000 °C. High strength and toughness plus a very good wear resistance of the cutting tool material are necessary. This internal test revealed that the SiC–SiAlON-composite (10 vol% SiC, CeramTec grade "SL506") has a higher wear resistance, that means lower wear than the SiAlON without any hard particles itself (Fig. 6). Unfortunately, the other composites with Ti(C,N), $MoSi_2$ or WC addition did not show any improvement. Considering the results of microstructure and hardness/toughness measurements, the Ti(C,N)-composites should have the highest wear resistance. However, the application tests revealed almost the opposite: 10 vol% Ti(C,N) had no significant effect on wear compared to the alpha/beta-SiAlON without any particle addition. Composites with 20% Ti(C,N) showed the highest wear of all tested samples. And 30% lead to severe chipping from the beginning and the test had to be stopped. Also the WC-composite showed a big wear mark after the test. By checking the phase content it was found out that the peaks and therefore the amount of tungsten carbide in this sample was much lower than that of the samples used for prior characterization, although the same sample preparation was carried out. Maybe sedimentation of the "heavy" WC particles led to a depletion of this compound in the samples prepared for the machining test. However, it can generally be assumed that in spite of the high hardness of this material its toughness is not enough for the interrupted cutting conditions. Most probable this material would show a higher wear resistance under "soft" cutting conditions. The $MoSi_2$-composites exhibited only poor mechanical properties. But unexpectedly this material did not show the expected catastrophic wear behaviour. Maybe the Mo_5Si_3 has an unknown positive effect during the machining process.

In summary, although the first attempt to prepare and test SiAlON-particulate composites other than SiAlON–SiC did not produce satisfying results, the authors expect that by further optimization materials with higher wear resistance can be developed.

4. Conclusions

SiAlON cutting materials with different reinforcing particles (SiC, $Ti(C_{30}N_{70})$, $MoSi_2$, WC) were prepared by gas pressure sintering. Their microstructure, properties and wear behaviour in a machining test were evaluated.

Under the sintering conditions a strong interaction of $MoSi_2$ and WC with the matrix was observed, resulting in a change of the composition of the reinforcing phase and also of the alpha/beta-SiAlON ratio. This strongly influenced the properties of the composites.

The best wear behaviour exhibited the material with 10 vol% SiC. The material reinforced with $MoSi_2$ showed a reasonable wear behaviour – despite its low fracture toughness and low hardness – indicating some special effects, which should be investigated in more detail.

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