

# **$\alpha$ - SiAlON Based Ceramic Cutting Tools for Carbon Fiber Reinforced Carbon Composites Machining.**

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## **Abstract**

*The substantial increase on carbon-carbon composites applications are related to its excellent ablation, thermal shock resistance, high stiffness, chemical inertia and low specific mass. However, manufacturing costs are still relatively high, including machining. New cutting tools are continuously being developed, aiming an increase at machining speed and cost reduction. In this work,  $\alpha$ -SiAlON based ceramic cutting tools were produced by pressureless sintering, using  $Al_2O_3$ ,  $Ce_2O_3$ ,  $Y_2O_3$  and  $AlN$  as additives. The sintered samples were 98,54% dense, with a hardness of 20 GPa and fracture toughness of  $6,5 \text{ MPa}\cdot\text{m}^{1/2}$ . Cutting tools of a geometry according ISO 1832 were produced, and some of them were coated by a hot filament-assisted Chemical Vapor Deposition (HFCVD) diamond coating process. The coated and uncoated cutting tools were tested in Carbon Fiber Reinforced Carbon (CFRP) composites machining, to evaluate the diamond coating influence on machining performance. After the tests, the uncoated tools presented severe flank wear and shorter life than the diamond coated ceramic tools. This flank wear is caused by the abrasive carbon powder generated during the facing operation.*

*Keywords: Ceramic; Cutting Tools; Composites; SiAlON;*

## **1. Introduction**

There is a significant increase on use of silicon nitride ( $Si_3N_4$ ) based ceramics as structural materials as a result of their excellent mechanical and chemical properties [1-2]. They are promising candidates for cutting tools and, therefore, have been intensively investigated in the last decade. [2-3]. One key issue to produce high quality and reliable structural  $Si_3N_4$  ceramics is to obtain high fracture toughness, which can be achieved by the combination of several factors such as: high quality starting powders, optimization of the processing parameters aiming to reduce defect size and frequency, production of high aspect ratio grains and control of the interfacial bonding strength between the  $\alpha$ - $Si_3N_4$  grains and the secondary intergranular phase or reinforcement phase [1-4].

Due to the low self-diffusion coefficients, it is common to use oxides as sintering additives such as  $Y_2O_3$ ,  $Al_2O_3$  or rare earth oxides, which form liquid intergranular phase at elevated temperatures to promote densification. However, the secondary intergranular phase is detrimental to the high temperature mechanical properties due to

softening. In this context, the use of specific mixtures of  $Y_2O_3$ ,  $Al_2O_3$  and AlN as additives forms SiAlONs, which are advantageous because they absorb a large part of the additives, forming a solid solution and leading to high temperature properties improvements [5].

The outstanding hardness of diamond is the reason for its increased application as coatings for cutting tools. The use of  $\alpha$ -SiAlONs based ceramics coated with diamond combines the substrate properties, such as fracture toughness and strength, with the diamond hardness.

Carbon Fiber Reinforced Carbon (CFRC) composites are widely employed in aerospace and nuclear industries, as a result of their excellent thermo-mechanical properties in non-oxidizing atmospheres, high chemical inertness and relatively low density [6]. Despite the near-net shape fabrication methods used for most fiber composites, machining operations are often employed, and need to be highly efficient to compensate elevated fabrication costs, increasing the productivity without quality losses. New cutting tools need to be developed to overcome the existing difficulties for machining such composites [7].

In this work,  $\alpha$ -SiAlONs cutting tools, uncoated (as sintered) and coated with CVD-diamond in a hot filament-assisted reactor, were evaluated on CFRC composites machining.

## 2. Experimental Procedure

### 2.1. Processing

Commercial silicon nitride powder,  $\alpha$ - $Si_3N_4$  (HCST Germany), aluminum oxide ( $Al_2O_3$ -ALCOA - Brazil), Cerium Oxide, ( $CeO_2$  - HCST), yttrium oxide, ( $Y_2O_3$  - HCST) and aluminum nitride (AlN - HCST) were used as starting powders. The samples were prepared by ball milling, for 6 hour, using ethanol as media and powder batches composed of 78.30 wt %  $Si_3N_4$ , 3.15 wt.%  $Y_2O_3$ , 1.00 wt%  $Al_2O_3$ , 3.15 wt %  $CeO_2$  and 14.4 wt % AlN. After mixing, the suspension was dried first in a rotary evaporator and subsequently in an oven at 90 °C for 5 h. Green bodies were produced by uniaxial cold press under 50 MPa and isostatic cold press under 300 MPa. Sintering was done in a furnace with graphite heating elements in nitrogen atmosphere at 1850 °C for 30 min (heating rate of 15 °C).

### 2.2. Characterization

The rule of mixtures was used for theoretical density estimation. The density after sintering was determined by the immersion method.

The existing phases on sintered samples were determined by an X-Ray diffraction analysis, using Cu-K $\alpha$  radiation, at angles  $2\theta$  ranging between 20 and 80° under a step width of 0.02°. Lattice parameter refinement of the phases was executed using the software Trieste 1.0. The  $\alpha$ -SiAlON and  $\beta$ - $Si_3N_4$  phase contents were determined by the method proposed by Gazzara *et al* [8]. For microstructural analysis by Scanning Electron Microscopy (SEM) the specimens were ground, polished, and the surface was etched by a molten 1:1 mixture of NaOH and KOH at 500 °C for 1 minute.

The hardness was determined by Vicker's indenter under an applied load of 20 N, for 30 s. For statistical reasons, 21 indentations were made per sample. The fracture toughness was determined by measuring the crack length created by the Vickers indentations. The  $K_{IC}$  was then calculated by the relation proposed by Evans *et al.*[27], valid for Palmqvist type cracks.

The sintered SiAlONs samples were machined to adjust the cutting angles, curvatures and parallel surfaces, to serve as cutting tools.

### 2.3 Diamond coating

Prior to the diamond deposition by the CVD process, the samples were cleaned with acetone and treated in an ultra-sound apparatus for 30 minutes in a suspension of diamond powder and hexane, to facilitate the film growth and homogeneity. In the CVD process a hot filament-assisted chemical vapor deposition was used, under a H<sub>2</sub>- flux of 100 s.c.c.m. and 2 s.c.c.m. of CH<sub>4</sub>. Each sample face was submitted to a 4 h period of diamond growth. For deposition, 7 filaments of 85 µm were used, submitted to a continuous current of 17.5 A and 38 V. The substrate temperature was approximately 780 °C, and the pressure in the reaction chamber 50 torr, leading to a diamond coating approximately 2.5 µm thick.

### 2.4 Machining tests

Aiming to evaluate the cutting tools and diamond coating performances, facing tests of composites were carried out on commercial 2D Carbon Fiber Reinforced Carbon (CFRC) discs of 310 mm diameter and 11 mm thickness, with an apparent density of 1.42 gcm<sup>-3</sup> and 7 vol. % porosity, containing 45 vol. % of fibers (KKarb Tipe A, Kaiser Aerotech). The experiments were conducted under dry conditions, using a CNC lathe.

The behavior of cutting tools was examined based on cutting speed (V<sub>c</sub>) and cutting length (L<sub>c</sub>). For the first facing test, the feed rate (f), cutting speed and depth of cut (a<sub>p</sub>) were the same, for both coated and uncoated tools, as defined on Table 1. Subsequently, for the diamond coated SiAlON, the cutting speed and cutting length have been increased to 1000 m/min and 7000 m respectively, considering that no flank wear was observed below those values. The evaluation of damages on cutting tools during CRFC facing was done by Scanning Electron Microscopy.

Table 1- Parameters of the machining tests.

Tools	V <sub>c</sub> (m/min)	f (mm/rot)	a <sub>p</sub> (mm)	L <sub>c</sub> (m)
SiAlON	600	0,1	1,0	600
SiAlON-CVD	600	0,1	1,0	7000
	800			
	1000			

V<sub>c</sub>= cutting speed.                      a<sub>p</sub>= depth of cut  
L<sub>c</sub>= cutting length.                      f= feed rate

## 3 Results and Discussion

### 3.1 Phase analysis and final density

After sintering, the cutting tools were ground to achieve the final geometries according ISO 1832. Figure 1 presents uncoated SiAlONs cutting tools, with final dimensions of 12 x 12 x 4.8 mm and chamfer of 0.25 x.20°.

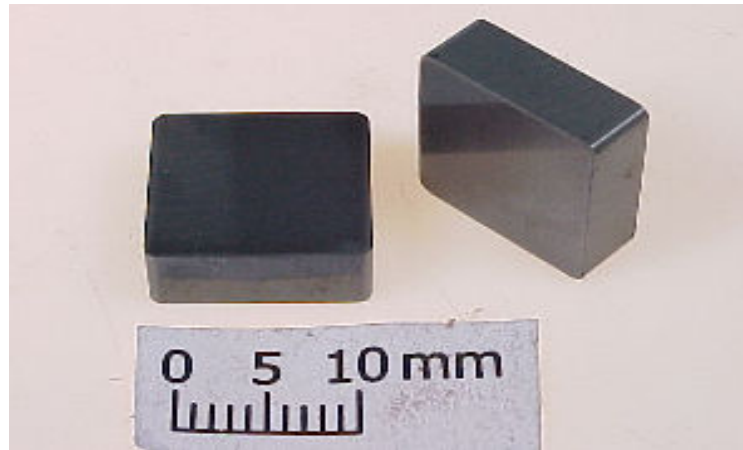


Figure 1 – General aspect of the uncoated SiAlON cutting tools.

The diffractometric phase analysis of the sintered specimens, Figure 2, revealed the presence of  $\alpha$ -SiAlON,  $\beta$ -Si<sub>3</sub>N<sub>4</sub> and small amount of AlN. A substantial amount of  $\alpha$ -SiAlON has been formed. The relative density of the samples after sintering was 98,54% of theoretical.

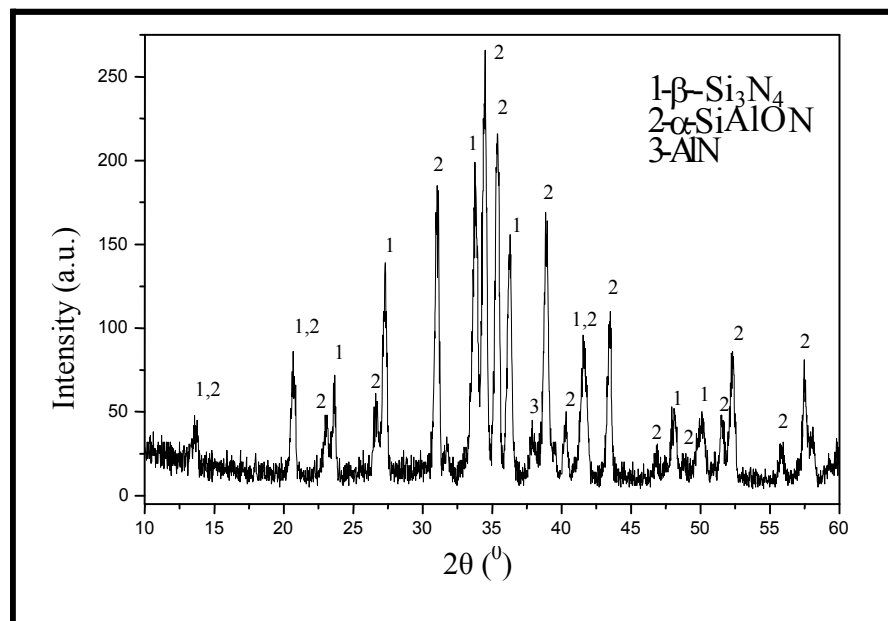


Figure 2 – X-ray diffraction pattern of the sintered specimens.

### 3.2 Microstructure

The microstructure of the etched sintered sample is shown in Fig.3.



Fig 3 – SEM micrograph of polished and etched surface of substrate.

During silicon nitride liquid-phase sintering,  $\alpha$ - $\text{Si}_3\text{N}_4$  particles are dissolved in the liquid phase and precipitated as elongated  $\beta$ - $\text{Si}_3\text{N}_4$  grains. The morphology, aspect ratio and grain size depends on the amount and type of additives used.

It is well known that the anisotropic growth of  $\beta$ - $\text{Si}_3\text{N}_4$  grains is related to the different growth rate on the basal plane and the lateral planes of the hexagonal  $\beta$ - $\text{Si}_3\text{N}_4$  prism [12]. The growth rate in the c-axis is higher than that in the a-axis, which leads to the anisotropic growth of these grains [10,11]. As reported in previous work, [4] samples sintered with high amounts of  $\text{Al}_2\text{O}_3$ - $\text{Y}_2\text{O}_3$  additives resulted in larger grains due to smaller degree of steric hindrance [12].

Figure 4 details CVD diamond film on  $\alpha$ - $\text{SiAlON}$  tool edge (4a). As can be seen in 4b, the diamond film was uniformly deposited on the substrate by the chosen CVD process.

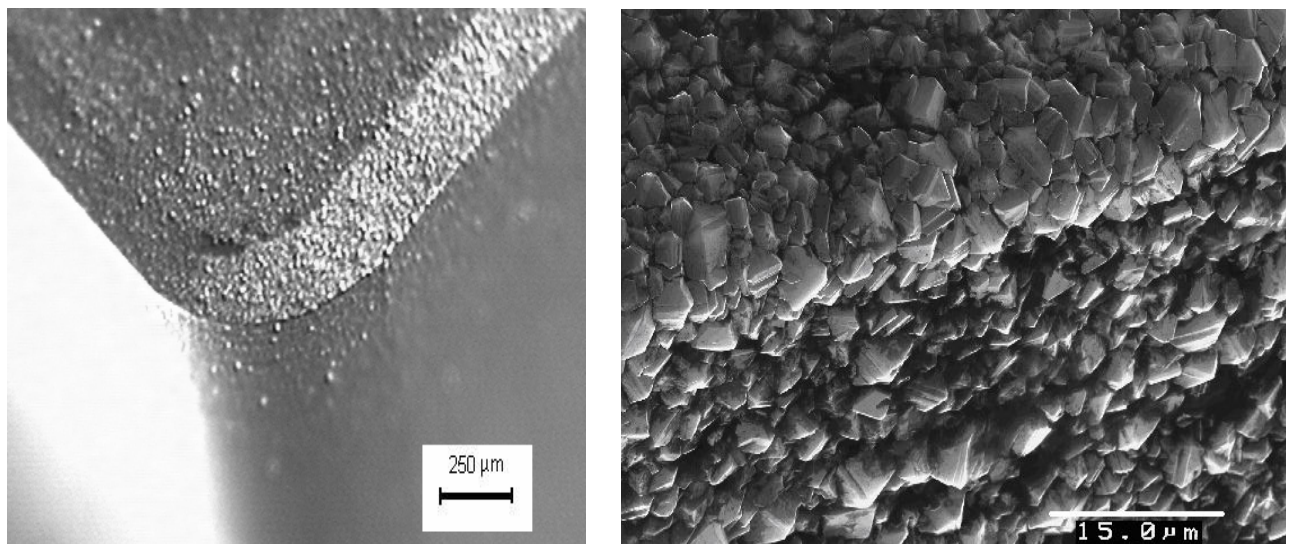


Figure 4 – SEM micrographs of the cutting tool: a) surface of the coated cutting tool edge, b) CVD diamond film.

### 3.3 Mechanical Properties

The average hardness of sintered ceramic cutting tool was 20 GPa, and this value is related to the high volume fraction of extremely hard  $\alpha$ -SiAlON particles. The high aspect ratio of the  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains resulted in a fracture toughness of 6.5 MPa.m<sup>1/2</sup>.

### 3.4 Machining tests

During machining of the carbon fiber reinforced carbon composites, large quantities of graphite powder are generated. The main acting wear mechanism of uncoated cutting tools are the abrasion and polishing of the flank face, generated by highly abrasive carbon powder originated from carbon fibers rupture and brittle fracture on carbon matrix. For this experiment on uncoated materials, greater flank wear is observed as cutting speed is increased. The Figure 5 show details of a cutting tools after machining.

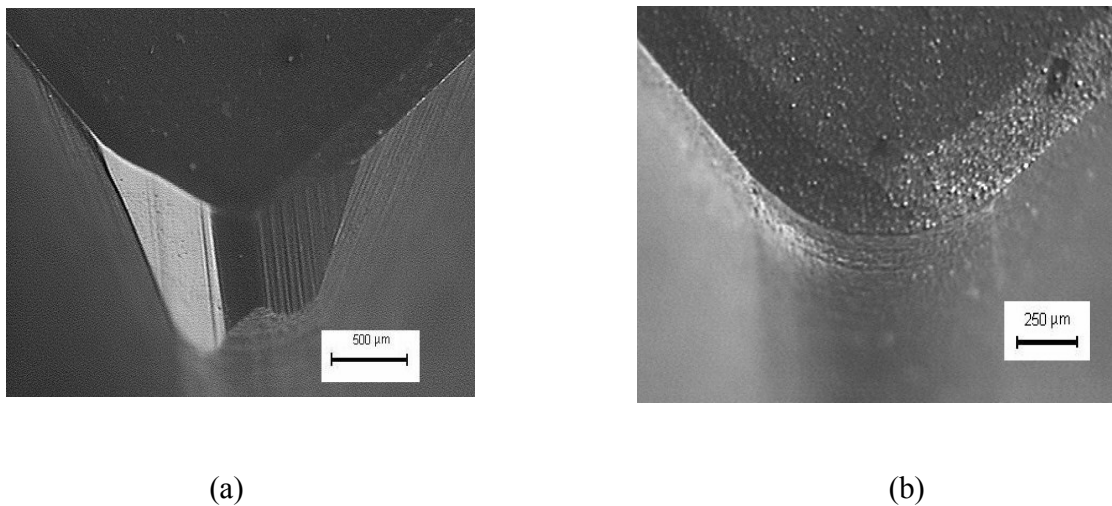


Figure 5: SEM micrographs of the cutting tools edges after machining to  $V_c=600$  m/min: a) uncoated cutting tool, b) diamond coated cutting tool.

For the diamond coated cutting tools, no flank wear is observed, even for higher cutting length and speed. The cutting edge remained unmodified. The CFRC facing operation reached 7000 m of cutting length, at 1000 m.s<sup>-1</sup>, and no cutting edge damage was identified, as can be seen on Figure 5b. The extremely hard CVD diamond coating allows the coated cutting tool to remain free of flank wear, even under severe test conditions. The very small carbon particles, originated from the machined composite, are not able to promote diamond film rupture, but instead, reduce its initial roughness and acts as a lubricant film during the facing operation. Therefore, when compared to the uncoated SiAlON cutting tools, the high wear resistance at high speed of the CVD coated tools allows CFRC machining on shorter periods, with greater tool-life and better quality of machined part.

### 3.5 Composite Surface Roughness

On CVD diamond coated SiAlON cutting tool, the machining promotes reduced average roughness on CFRC surface ( $6,4 \mu\text{m}$ ), when compared to uncoated tools ( $9,8 \mu\text{m}$ ). The cutting edge geometry for those tools is preserved. SEM observations revealed carbon particles adhered to diamond film after facing operation. For higher speeds, such particles form the lubricant film earlier, reducing the average roughness of the machined part.

For the uncoated tool, the excessive flank wear caused by abrasive carbon powder increases contact area between cutting tool and machined composite, raising rubbing and, as a consequence, the average roughness of machined composite. For the CFRC machined with uncoated coating tools, a greater amount of micro cracks was observed along the carbon matrix surface, as shown on Figure 6a. The high flank wear observed in this case, causing the cutting edge loss, promotes partial composite removal, favoring crack formation. For the coated samples, negligible cracks were detected (figure 6 b).

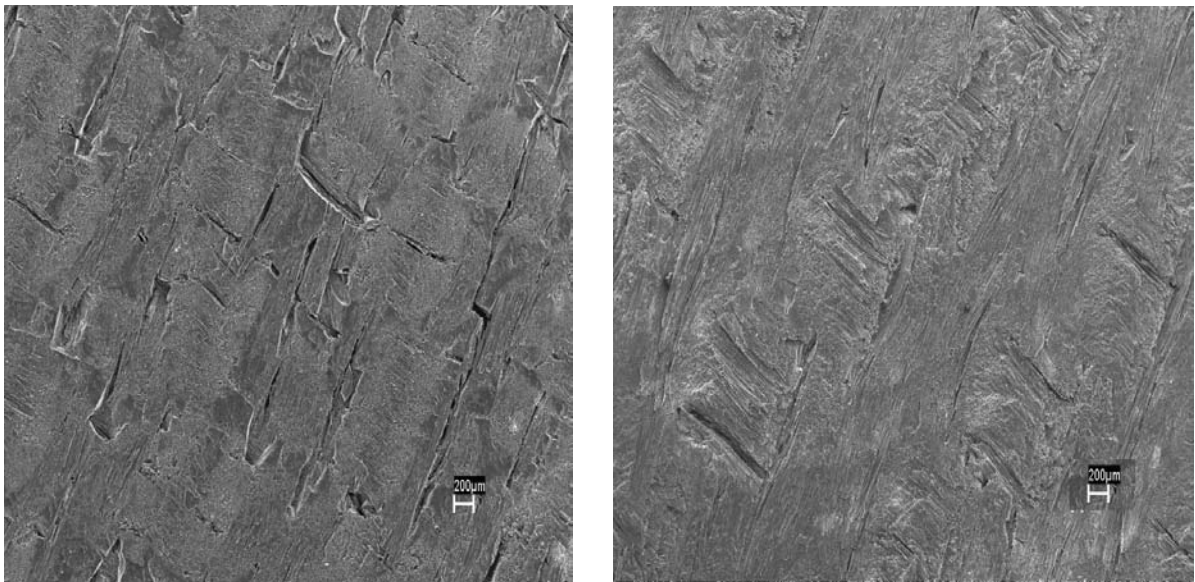


Figure 6: Composite machined surface: a) using uncoated tool, b) using diamond coated tool

### Conclusion

In this work a new class of  $\alpha$ -SiAlON based cutting tools has been developed. The association of  $\alpha$ -SiAlON and  $\beta$ -Si<sub>3</sub>N<sub>4</sub> resulted in a ceramic material with good fracture toughness of  $6.5 \text{ MPa}\cdot\text{m}^{1/2}$  and elevated hardness of 20 GPa. The machining tests of Carbon Fiber Reinforced Carbon composites indicated large differences on performance, for coated and uncoated cutting tools. For the uncoated  $\alpha$ -SiAlON based ceramics, high flank wear occurs, even for low cutting length. The wear is generated by highly abrasive carbon powder, originated from carbon fibers rupture and brittle fracture on carbon matrix. In this case, the average roughness of the machined composite is high. On CVD diamond coated  $\alpha$ -SiAlON ceramic tools, no flank wear was observed, and the cutting edge remained unmodified, even for severe test conditions, such as high cutting length and speed. Carbon particles, originated from the machined composite, do not promote diamond film rupture, but instead, acts as lubricant film and reduces composite surface

initial roughness The elevated wear resistance of CVD diamond coated  $\alpha$ -SiAlON ceramics makes this material adequate for high speed machining of Carbon Fiber Reinforced Carbon composites.

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