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Novel Composite cBN-TiN Coating Deposition Method: Structure and Performance In Metal Cutting

W.C. Russell, A.P. Malshe*, S.N. Yedave*, W.D. Brown*

Valenite Inc., Troy, Michigan, USA
*MRL-MEEG, University of Arkansas, Fayetteville. Arkansas, USA

Summary:

Cubic boron nitride coatings are under development for a variety of applications but stabilization of the pure cBN form and adhesion of films deposited by PVD and ion-based methods has been difficult. An alternative method for depositing a composite cBN-TiN film has been developed for wear related applications. The coating is deposited in a two-stage process utilizing ESC (electrostatic spray coating) and CVI (chemical vapor infiltration). Fully dense films of cBN particles evenly dispersed in a continuous TiN matrix have been developed. Testing in metal cutting has shown an increase in tool life (turning – 4340 steel) of three to seven times, depending on machining parameters, in comparison with CVD deposited TiN films.

Keywords:

Cubic boron nitride, cBN, titanium nitride, TiN, electrostatic spray coating, ESC, chemical vapor infiltration, CVI, composite coating

1. Introduction:

Diamond is the hardest known material and likewise the hardest material used in the cutting tool industry. However, since carbon is soluble in iron, diamond does not hold up well in applications on ferrous materials where significant cutting temperatures are generated. Cubic boron nitride (cBN), known as the second hardest material, has good thermal stability and chemical inertness with respect to ferrous materials. Currently, cBN is being used in it's bulk form as tool insert bodies or as brazed segments in combination with tool bodies, usually WC-Co. A number of technical groups around the world are also working on developing a cBN coating much in the way that diamond coatings have been developed for cutting tools in recent years.[1,2] Such coatings on cemented carbide substrates would provide the

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hardness and chemical resistance of cBN in combination with the toughness of a cemented carbide body.

Cubic boron nitride cutting tools are currently manufactured using conventional high pressure/high temperature forming technology. The resulting polycrystalline cBN (PCBN) compacts, due to their structural rigidity and limitations of the forming methods, cannot be manufactured in complex geometries such as chip-breaking cutting tools, rotary tools and other shapes. Cubic boron nitride coating technology would allow the incorporation of such complex shapes provided the coating technique is flexible enough. Such coated tools would be a significant product for surface finishing applications in the ferrous metal machining industry and would complement the PCBN compact tools used for coarse machining.

Most groups working on cBN film deposition employ either high energy plasma or ion bombardment to create stabilization of the cubic phase relative to the hexagonal or amorphous phases that are more thermodynamically stable but do not have the hardness of cBN.[3] These methods, although resulting in the formation of very thin films (< 1μm), of cBN have so far not demonstrated the capability to form cBN independent of the other phases.[4] Invariably, amorphous and/or hexagonal phase cBN forms first on the substrate before the cBN phase can be stabilized.[5,6] Another problem is the high intrinsic compressive stress associated with the cBN film when formed by these methods.[7,8,9] These stresses can overwhelm the adhesive bonds between the coating and the substrate leading to delamination and severely limits the functional thickness of these films.

Today the major roadblock to the realization of cBN coated cutting tools is the inability of conventional vapor deposition techniques to coat the cBN phase and at useful thicknesses. The challenges in the growth process are:

1) excess compressive stress
2) homogeneous phase synthesis
3) uniform stoichiometry control
4) thick coatings (> 1μm)
5) adhesion with substrate
6) phase stabilization on different substrates

These issues are magnified when considering manufacturing on an industrial scale.

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To overcome these problems, we feel the coating community will have to develop cBN films in combination with other coatings to resolve the phase stability and intrinsic stress issues. The result will be composite films with physical properties dependent on the material makeup of the system.

The approach reported here is an alternative method to reach the same end of a composite film containing cBN. Instead of attempting to grow cBN from the vapor phase, however, we start with fine sub-micron cBN powder and apply it as a coating. We then use modified conventional coating methods to fix the cBN particles in a matrix and bind them with excellent adhesion to the substrate.

2. Experimental Approach:

This project deals with the development of a novel hybrid technology involving electrostatic spray coating (ESC)[10,11] followed by chemical vapor infiltration (CVI).[12] This NSF supported GOALI program has been a collaboration between the Materials and Manufacturing Research Laboratories (MRL), University of Arkansas and the Materials Research and Development (MRD) division of Valenite.

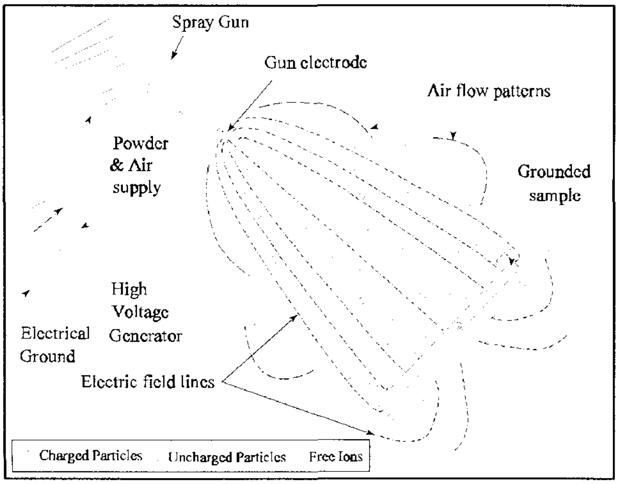

Fig. 1: Schematic of ESC process.

In the electrostatic spray coating (ESC) process the powder particles are generally electrically insulating in nature and can carry the static charge over

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a distance of a few tens of centimeters. The electrostatic charge is generated on the powder particles (cBN) which are fed into an electrostatic spray gun that typically generates a charge of a few tens of kilovolts. When ejected from the spray gun the positively charged powder particles follow the electric field lines toward the grounded objects (substrates) and coats them (Fig. 1).

Electrostatic spray coating process variables were optimized using a matrix of experiments as described in Yedave et al.[13] On the surface of a tool the cBN particles deposited by ESC have the appearance in cross section as shown in Figure 2.

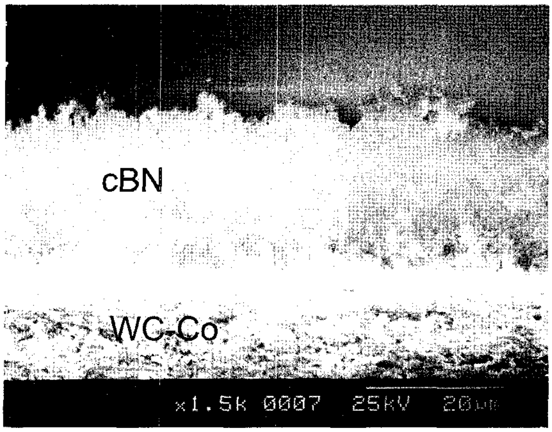

Fig. 2: SEM cross-section of ESC deposited cBN (1500X).

There is no issue with particles stacking on top of one another and as shown in Fig. 2, they do so very uniformly so that a surprisingly smooth layer of particles can be deposited across the surface of the tool. However, these particles are only loosely bound (van der Waals forces) to each other and to the substrate.

Chemical vapor infiltration (CVI) is a process that is used to convert the ESC treated surface into a hard composite coating, strongly bound to the substrate surface. This process is used in many applications in various industries but was developed by Valenite MRD for this application. CVI is closely related to chemical vapor deposition (CVD) and the same equipment is used for each process. The deposition of titanium nitride (TiN) is controlled in CVI in such a way that the TiN coating grows on the individual cBN particles and substrate uniformly throughout the depth of the ESC layer. This can be done by

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manipulating CVD parameters for the deposition of TiN so that the rate of diffusion of the reactants, $TiCl_4$, H_2 and N_2, is faster than the rate of growth of the TiN layer. In other words, the reactant diffusion rate must be faster than the rate of formation of the products, TiN and HCl. This can be accomplished by slowing down the reaction:

$$2TiCl_4 + 4H_2 + N_2 \rightarrow 2TiN + 8HCl \quad (1)$$

It is well known that this reaction is surface reaction rate controlled and that the kinetics are temperature sensitive. However, it is difficult to slow the reaction down, by reducing temperature uniformly in a large reactor vessel. Instead, an alternative chemical approach was used in which one of the products, HCl, was added along with the reactants to shift the kinetics of the reaction toward the reactants. This proved to be very effective in slowing the reaction uniformly to a desired rate. As a result, all the void space existing in the ESC coating was filled 100% with TiN coating. TiN is also a good choice in that it contacts or "wets" the surfaces of the cBN particles extremely well leaving no apparent gaps or porosity. It is well documented that CVD-TiN coatings have excellent adhesion when appropriately applied to WC-Co surfaces. The contact between the cBN and TiN phases is very important in that the TiN matrix must effectively hold the cBN in place during metal cutting so that the cBN phase can be functional. Also, of course, the whole composite coating system must be securely bound to the substrate surface to function. Other CVD-based coatings can likewise be used to create other composite coating systems with varying material properties. Once the CVI process is complete the tools can be handled.

A cross section of a cBN-ESC followed by TiN-CVI treated tool is shown in Figure 3. Notice the uniform dispersion of cBN particles and complete penetration of the TiN phase. Also notice that the TiN coating can be continued once the pore volume filling is complete to essentially establish a pure TiN layer on top of the composite layer. This can be useful for reducing roughness caused by the conformality of the coating on the cBN particles.

Since both ESC and CVI are not line-of-sight processes, they can be combined to coat parts of complex shape, geometry or multiple faced tools.

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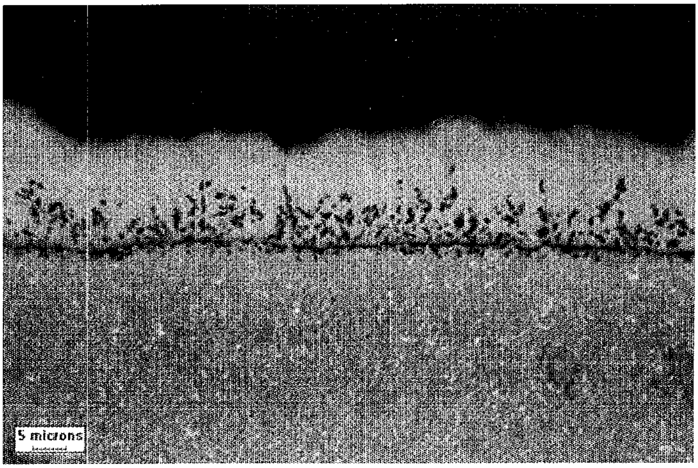

Fig. 3: Cross-section of TiN infiltrated cBN coating.

3. Testing and Evaluation:

Composite cBN-TiN coated WC-Co tools were tested for coating uniformity, adhesion and in the machining of steel. Adjustments to the ESC process were primarily responsible for improving uniformity as development progressed. The result is a continuous coating as shown in Figure 3. There is some roughness as compared with normal CVD coatings due to the presence of the cBN particles, which have an average diameter of approximately one micron or less. Particles in clusters are actually separated due to the growth of TiN around the individual particles. However, the system of particles becomes fixed in place as the growing TiN coatings link and become a continuous matrix and the gaps are filled in. The degree of roughness can be reduced, as mentioned above, by coating above the range of the cBN.

Adhesion was measured using a CSEM Revetest® automatic scratch tester fitted with an 80° diamond. Lateral table speed used was 10 mm per minute and the force application rate was 100 newtons per minute. Acoustic response curves were generated for a composite coating and a pure TiN coating. The critical load was the same for both coatings indicating that the presence of cBN particles at the tool surface does not have a deletereous effect on the bond that is established between the TiN coating and the substrate. Contact surface area is not even reduced since the TiN grows

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uniformly all the way around the cBN particles, lifting them slightly from the tool surface until they become fixed in place by the TiN matrix. This results in 100% contact between the TiN and WC-Co phases with no contact between WC-Co and cBN. This is the ideal situation for maximizing adhesion since it parallels that of the pure TiN coating. Also, no cohesive failure was observed within the coating as a result of the scratch test. This indicates good bonding within the coating and most notably between the TiN and cBN phases.

For machine testing we decided to apply the coating in a turning operation (4340 steel, Rc 28-30, 600 sfm, 0.010 ipr, 0.08" DOC, CNGA432, dry). A typical 6% cobalt substrate grade was utilized. A pure TiN film was used to benchmark performance. The results are shown in Figures 4, 5 and 6.

Although the composite coatings were rougher than their TiN counterparts, the starting point and progression of wear was consistantly significantly lower for the composite tools. These and other similar tests indicated that composite inserts had tool lives in the range of three to seven times that of regular CVD TiN coated tools. When we studied the wear areas we observed that the cBN particles were being held rigidly in place by the TiN matrix even where wear was most severe. The SEM photo in Figure 7 shows an area that has been subjected to abrasive wear. The cBN particles are the small dark spots distributed evenly throughout. Notice that the particles, although exposed on the wear surface are still held in place by the TiN matrix and that wear is occuring evenly across this area. This indicates that the TiN is doing its job in holding the cBN in place and to the surface of the tool while the cBN is doing its job as evidenced by the wear progression graphs shown in the following figures.

We have demonstrated this kind of performance both with flat inserts and with tools designed with chipbreakers. Chipbreakers are not an option with traditional PCBN tools.

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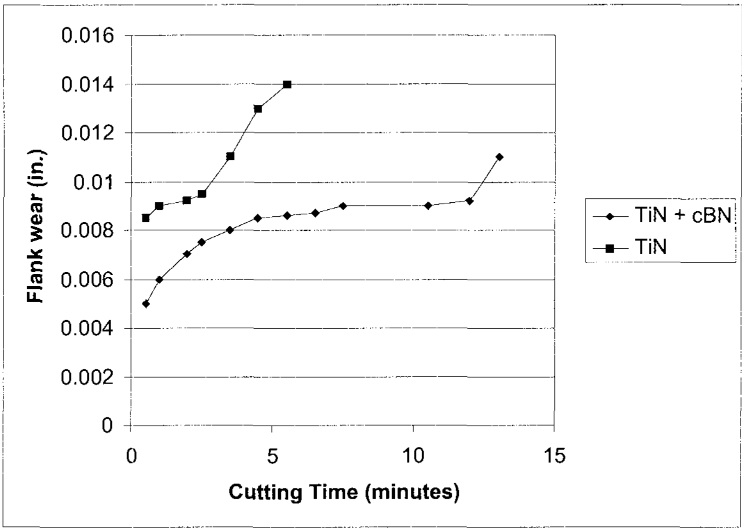

Fig. 4: Flank wear, Turning 4340 Steel, Rc 28-30 (600 sfm, 0.010 ipr, 0.08 in. DOC, CNGA432, dry machining)

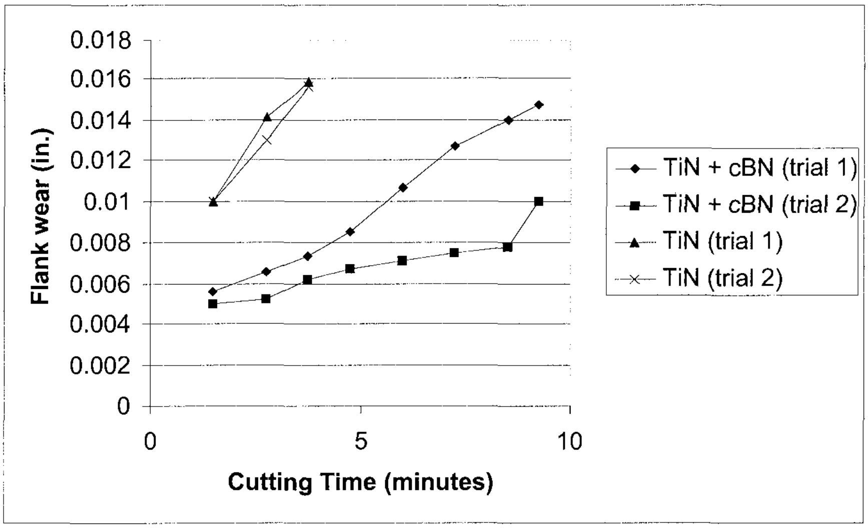

Fig. 5: Flank wear, Turning 4340 Steel, Rc 28-30 (600 sfm, 0.010 ipr, 0.08 in. DOC, CNGA432, dry machining)

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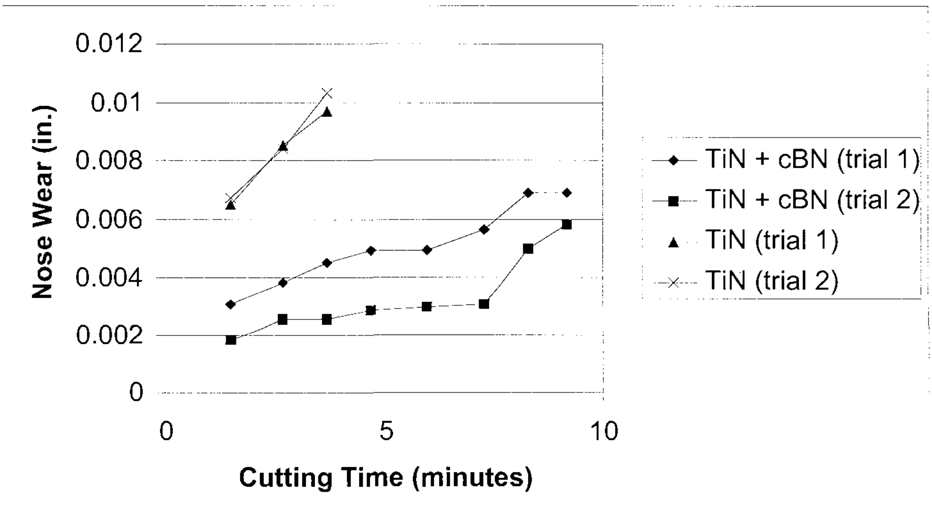

Fig. 6: Nose wear, Turning 4340 Steel, Rc 28-30 (600 sfm, 0.010 ipr, 0.08 in. DOC, CNGA432, dry machining)

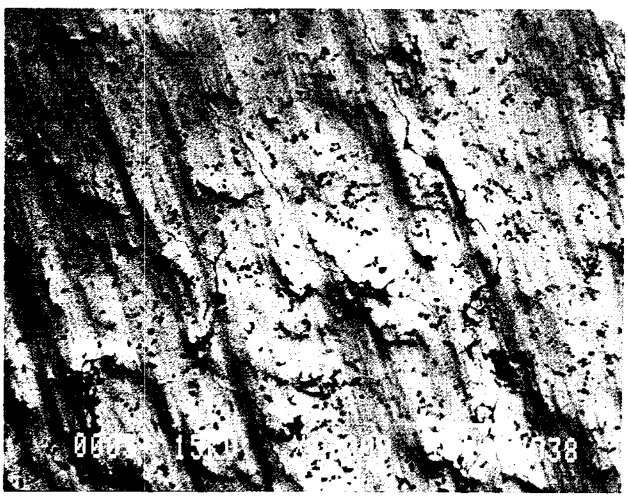

Fig. 7: SEM micrograph of wear zone in cBN-TiN coating. Dark particles are cBN in a TiN (gray) matrix (1000X).

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3.1 Future Testing:

The next step will be to benchmark against PCBN tools. PCBN also contains a binder material, a good percentage of which is also usually TiN. However, the cBN/TiN ratio is lower (30-45% cBN) for our coating than it is for the PCBN systems.

We also plan to test against other CVD coatings since the comparison with TiN is so dramatic. Normally, a tool life improvement of this magnitude (3-7 times) is not observed through design improvements of CVD coatings.

4. Technology Application:

What is the application range for cBN-TiN composite coated carbide relative to standard coated carbide, ceramic and cBN tools? Not enough testing has been done to answer this question precisely but we do know enough based on physical properties and tests measured so far to make estimations. We anticipate that a standard speed and feed diagram for these products will look approximately like the graph shown in Figure 8.

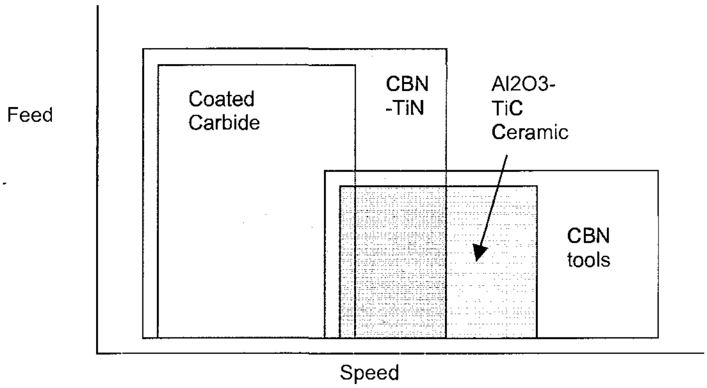

Fig. 8: Feed/speed diagram of anticipated performance range for composite cBN-TiN films on cemented carbide tools.

CBN composite tools will likely essentailly overlap coated carbide tools as they are known today, but also extend further into high speed areas where ceramics are used. There will be limitations at higher speeds (finer cuts) on

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materials above 55 Rockwell "C" hardness where pure ceramic tools will still have to be used. Composite coatings will be able to be used in applications where improved wear resistance is needed (nose and flank wear) above that offered by CVD coatings.

Composite cBN-TiN coatings will be able to be used in place of ceramic or cBN tools where a combination of toughness and hardness is required. Coated carbide does not have sufficient wear resistance and ceramics are too brittle. Also, since the coating can be applied to chipbreaker forms, it can be used in combination with tool geometries that are not available with ceramic tools to help control chip formation and cutting forces.

Cubic boron nitride business worldwide is about $160 M (US). About half of this is in the automotive sector. The growth rate for cBN tools has been 14% per year.

In the area of hard turning, where turning operations are substituted for grinding, about half the market would benefit from chipbreaking technology although right now this is currently unavailable. Cut this in half again for the market that would be addressable with this technology.

Abrasive metal machining will also be addressed with this technology. Using positive tool designs, also currently unavailable, would be advantageous.

5. Summary:

Cubic boron nitride based composite coatings have been developed by combining the techniques of ESC and CVI. ESC is used to deposit a uniform layer of ≤1μm diameter cBN particles on a tool surface. CVI is subsequently used to fix the particles in place and to the tool surface. TiN is the matrix coating that has been used but other CVD coatings can be applied (TiC, TiCN, Al_2O_3). In the current state of the technology the composite coatings have been shown to have good uniformity, except for micro-roughness associated with the cBN particles, good adhesion and improved machining performance in steel turning relative to pure CVD-TiN coatings. Potential application areas are hard turning, abrasive metal machining and other areas where CVD coatings do not currently supply enough abrasive wear resistance or where cBN and ceramic tools are too brittle or do not allow significant chip control or control of cutting forces.

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