

New Coating Technology “Nano-Lamellae CVD-AITiN”

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Ceramic coatings adopted to cutting tools include chemical vapor deposition (CVD) coating and physical vapor deposition (PVD) coating. The CVD coating has excellent wear resistance and heat resistance, but is inferior in toughness to the PVD coating. By controlling the coating structure on the nanometer order using the CVD method, we have established a new CVD coating technology that ensures the toughness of PVD coatings while maintaining the wear resistance and heat resistance of conventional CVD coatings, and enabled its mass production. This paper reports on its unique structure, excellent physical properties, and outstanding cutting performance when applied to cutting tools.

Keywords: CVD, TiAlN, cutting tool, high-speed machining, high-efficiency machining

1. Introduction

Cutting tools are used to machine various mechanical and metal parts including automotive parts. Cutting tool materials include high-speed tool steel, cemented carbide*¹, cermet, ceramics, cubic boron nitride (cBN), and diamond. These materials are adopted appropriately according to cutting speed and machining precision. Among them, the most frequently used is cemented carbide due to its superb balance between wear resistance and fracture resistance. Specifically, cemented carbide tools provided with a vapor-phase-coated thin ceramic film on their surfaces exhibit both the toughness of the substrate, i.e. cemented carbide, and the high heat and wear resistance of ceramic coating. Consequently, coated cemented carbide tools enable high-efficiency machining at a high speed and a high feed rate, resulting in reduced machining costs at the user end. The proportion of their usage has increased each year. Figure 1 presents a production breakdown, by tool grade, of indexable inserts (in Japan) and shipped quantities (statistics provided by the Japan Cutting & Wear-resistant Tool Association). As shown in the figure, coated cemented carbide tools account for approximately 70% of indexable inserts.

Methods used to coat a thin ceramic film onto a cutting tool include chemical vapor deposition (CVD) and physical vapor deposition (PVD). Table 1 summarizes the features attributable to differences in manufacturing method and principal uses of CVD and PVD for coating cemented carbide tools. Compared with PVD, CVD is higher in adhesion strength between the coating and substrate, making it easier to achieve a thick film. Therefore, CVD provides higher resistance to heat and wear than PVD. Conversely, CVD, which deposits films at a high temperature close to 1,000°C, produces residual tensile stresses within the film due to a difference in thermal expansion coefficient between the substrate cemented carbide and thin ceramic film, resulting in reduced strength. Accordingly, CVD is inferior to PVD in terms of chipping resistance. Various approaches have been used to improve CVD to overcome this drawback.⁽¹⁾ However, no approach has been successful in overcoming the cutting performance tradeoff between the two methods.

Against this backdrop, applying its advanced nano-technology and proprietary CVD coating technology, Sumitomo Electric Industries, Ltd. has developed a new

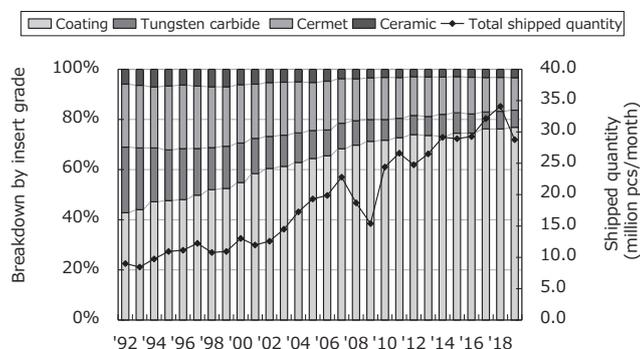


Fig. 1. Indexable insert shipment breakdown by grade, and shipped quantities (Japan)

Table 1. Comparison between CVD method and PVD method

	CVD (Chemical Vapor Deposition)	PVD (Physical Vapor Deposition)
Principle	With reactive gas at a high temperature (thermal CVD)	Ionization of vaporized metal atoms (Arc, Ion beam, Sputter etc.)
Film material	TiC, TiN, TiCN, Al ₂ O ₃ (equilibrium)	TiC, TiN, TiCN, TiAlN, CrN (non-equilibrium)
Coating temperature	800 - 1000°C	400 - 600°C
Adhesion	Excellent	Good
Stress	Tensile (approx. 1 GPa)	Compressive (approx. -2 GPa)
Strength (TRS* ²)	Failure	Good
Typical film thickness	5 - 20 μm	0.5 - 5 μm
Application	Turning insert (continuous cutting, insulation efficiency), Milling insert	Milling insert, Endmill (interrupted cutting, sharp edge), Drill

CVD coating termed “Nano-Lamellae CVD-AlTiN,” which offers chipping resistance comparable to that of PVD coatings, while retaining the wear resistance of CVD coatings. This paper reports on the properties and cutting performance of the new CVD coating.

2. Features of Nano-Lamellae CVD-AlTiN

Titanium aluminum nitride ($\text{Al}_x\text{Ti}_{1-x}\text{N}$) is in general use as thin ceramic films for cutting tools because it is very hard and exhibits high resistance to heat and oxidation.⁽²⁾ $\text{Al}_x\text{Ti}_{1-x}\text{N}$ exhibits two crystalline structures: cubic NaCl type structure ($c\text{-Al}_x\text{Ti}_{1-x}\text{N}$) and hexagonal wurtzite structure ($h\text{-Al}_x\text{Ti}_{1-x}\text{N}$). The former, $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$, is higher in hardness and is therefore suitable for forming thin ceramic films for cutting tools. However, at coating temperatures above 600°C , the proportion of $h\text{-Al}_x\text{Ti}_{1-x}\text{N}$ sharply increases.⁽³⁾ Therefore, PVD is used to deposit $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ films. Moreover, while $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ increases in hardness with an increasing amount of Al in the film, transition of crystal structure to $h\text{-Al}_x\text{Ti}_{1-x}\text{N}$ is initiated when the amount of Al exceeds 60 mol% ($x > 0.6$), leading to a sharp decrease in hardness.⁽³⁾ Sumitomo Electric has engaged in extensive research and development on high-Al-content $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ with the aim of applying it to cemented carbide tools. The Company has achieved deposition of a high-Al-content $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ film through CVD, in place of PVD, in a high vacuum using extremely reactive ammonia (NH_3) as a source gas. It has also had success in applying the method to mass production as Nano-Lamellae CVD-AlTiN (hereinafter, “CVD-AlTiN”), which has the following features.

Photo 1 shows a cross section of a CVD-AlTiN coating observed with a scanning electron microscope (SEM). CVD-AlTiN has a dense columnar structure. More specifically, it is an extremely fine structure with its average grain size being approximately $0.3\ \mu\text{m}$. Photos 2 and 3 present cross sections of a CVD-AlTiN coating observed with a transmission electron microscope (TEM). As shown in the electron diffraction image in Photo 2, the crystal grain of CVD-AlTiN, which forms a columnar crystal structure, exhibits a type-B1 (NaCl type) electron diffraction pattern. It shows NaCl type structure ($c\text{-Al}_x\text{Ti}_{1-x}\text{N}$).

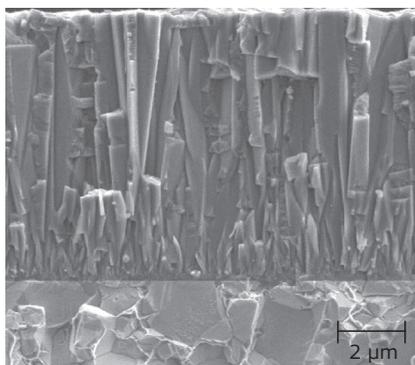


Photo 1. Cross-sectional structure of Nano-Lamellae CVD-AlTiN imaged with SEM

Photo 3 captures a cross section of a CVD-AlTiN coating observed with TEM at a higher magnification. As presented in Photo 3 (a), light and dark contrast is present in the crystal grains of CVD-AlTiN. A further magnified observation reveals alternate laminations of a light layer and a dark layer at intervals of approximately 5 nm, as presented in Photo 3 (b). In the direction crossing the light and dark layers, which is along the white line in Photo 3 (b), a line analysis was conducted using energy dispersive X-ray spectroscopy (EDX). Figure 2 presents the analysis results. The EDX analysis detected the elements Ti, Al, and N constituting CVD-AlTiN. Of these elements, N was almost constant irrespective of measurement position, while Al and Ti exhibited modulation in opposite phase with respect to each other. These results reveal that the alternate lamination of two regions forms CVD-AlTiN, where the light and dark areas in Photo 3 (b) are the Al-rich region with a high content of the light element Al and the Ti-rich region with a high content of the heavy element Ti, respectively. For the mechanism acting in the formation of the alternate lamination, it is likely that $\text{Al}_x\text{Ti}_{1-x}\text{N}$, which is in a metastable phase at high temperatures, undergoes spinodal decomposi-

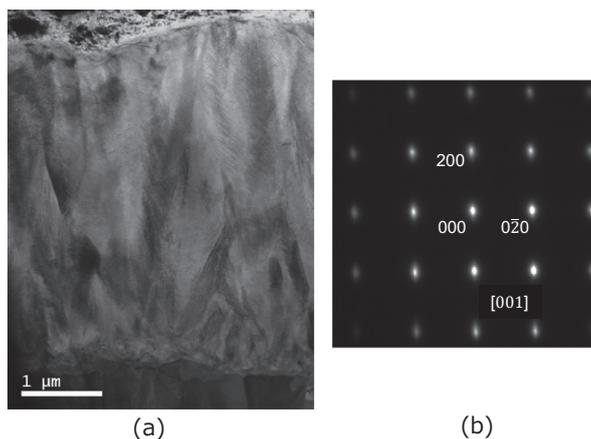


Photo 2. Cross section of Nano-Lamellae CVD-AlTiN (a) Structure imaged with TEM and (b) Electron diffraction pattern

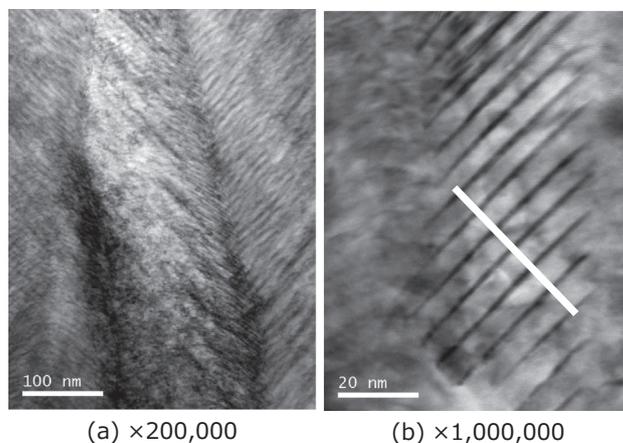


Photo 3. Cross-sectional structure of Nano-Lamellae CVD-AlTiN imaged with TEM (a) $\times 200,000$ and (b) $\times 1,000,000$

tion*³ in the process of cooling down to room temperature. Figure 2 also plots the atomic ratio of Al to (Al + Ti) determined based on the EDX analysis results. The ratio x is 0.9 at the maximum, with the average being 0.82, revealing the formation of highly Al-rich $Al_xTi_{1-x}N$. Notwithstanding this Al-rich composition, CVD-AlTiN retains a cubic NaCl type structure. This is probably due to stabilization brought about by the above-mentioned spinodal decomposition.

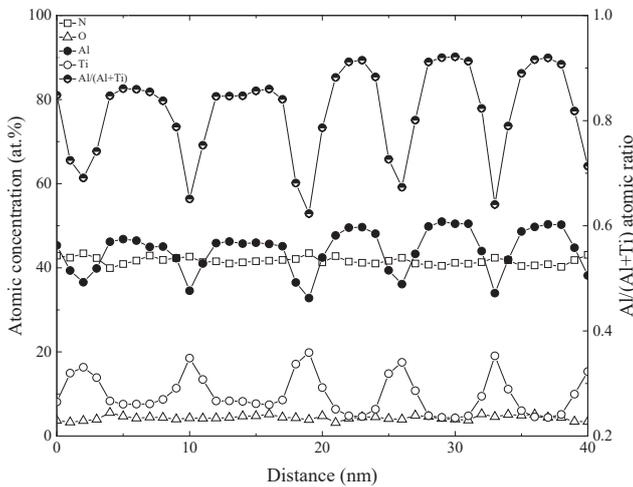


Fig. 2. Elemental analysis of CVD-AlTiN conducted with TEM-EDX

Figure 3 shows the hardness and Young's modulus of CVD-AlTiN coating measured by a nanoindentation method. In the measurement, a test load of 19.6 mN with respect to the diagonal cross section of the film was used for evaluation. Moreover, for comparison purposes, Al_2O_3 deposited through CVD (hereinafter, "CVD- Al_2O_3 ") was also measured on the same occasion. The hardness of CVD-AlTiN was 34.5 GPa, approximately 20% higher than the 28.7 GPa of CVD- Al_2O_3 . The Young's modulus of CVD-AlTiN was 624 GPa, approximately 30% higher than 471 GPa of CVD- Al_2O_3 . Consequently, CVD-AlTiN has higher hardness and strength than conventional CVD- Al_2O_3 .

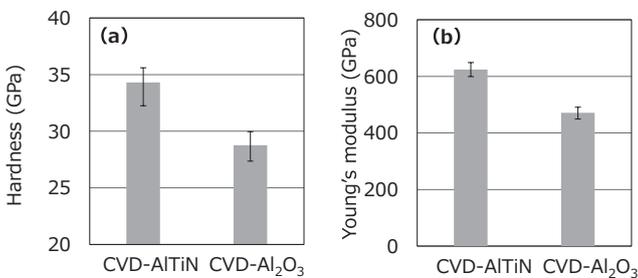


Fig. 3. Nanoindentation hardness (a) and Young's modulus (b) of CVD-AlTiN

Residual stresses in CVD-AlTiN were measured by a thin film X-ray method. Figure 4 shows the measurement results. In as-deposited CVD-AlTiN on a cemented carbide substrate, a tensile residual stress of approximately 1.8 GPa is present, as illustrated in Fig. 4. Tensile residual stresses present in a ceramic film have adverse effects on the film's toughness. For example, such stresses facilitate the growth of microcracks generated during cutting by contact impact with workpieces, resulting in tool edge chipping. To remove the residual tensile stress and introduce a compressive stress, CVD-AlTiN after deposition was subjected to shot peening treatment*⁴. "After treatment" in Fig. 4 indicates the results of shot peening and reveals a residual compressive stress of approximately 2.0 GPa introduced in the film through shot peening. The residual stress in a PVD-deposited AlTiN film measured for comparison purposes was approximately 2.6 GPa. Accordingly, owing to shot peening, CVD-AlTiN develops residual compressive stresses comparative to those in PVD-AlTiN.

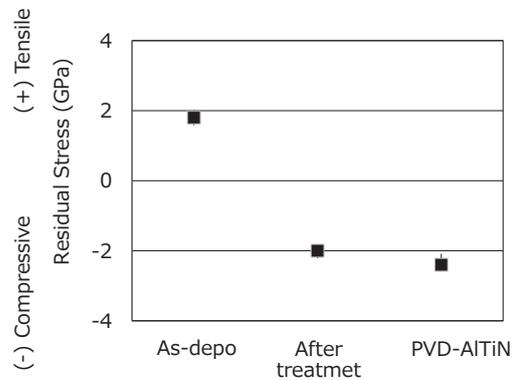


Fig. 4. Measurement results for stresses in CVD-AlTiN

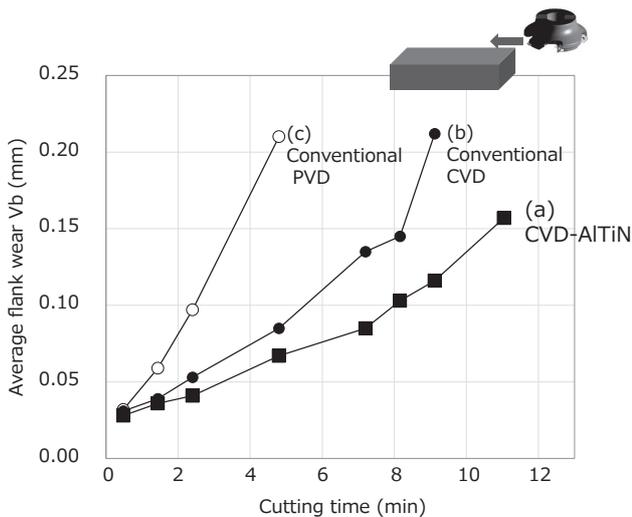
3. Performance of Nano-Lamellae CVD-AlTiN

CVD-AlTiN with the aforementioned features was applied to cutting tools. Its performance evaluation results are described below.

Figure 5 presents the wear resistance evaluation results for CVD-AlTiN. FCD700 blocks were used as workpieces. The cutting conditions were: Cutting speed (V_c) = 150 m/min, Feed per tooth (f_z) = 0.20 mm/t, Radial depth of cut (a_e) = 80 mm, Axial depth of cut (a_p) = 2.0 mm, and dry condition (Dry) without using a cutting fluid. Workpieces were cut until: Average flank wear (V_b) > 0.15 mm. CVD-coated TiCN/ α - Al_2O_3 (hereinafter, the "conventional CVD") and PVD-coated AlTiN (hereinafter, the "conventional PVD") were also used to cut workpieces on the same occasion for comparison purposes. Under these conditions, CVD-AlTiN proved its superb resistance to heat and wear, improving tool life to about three times and 1.3 times that of the conventional PVD and conventional CVD, respectively.

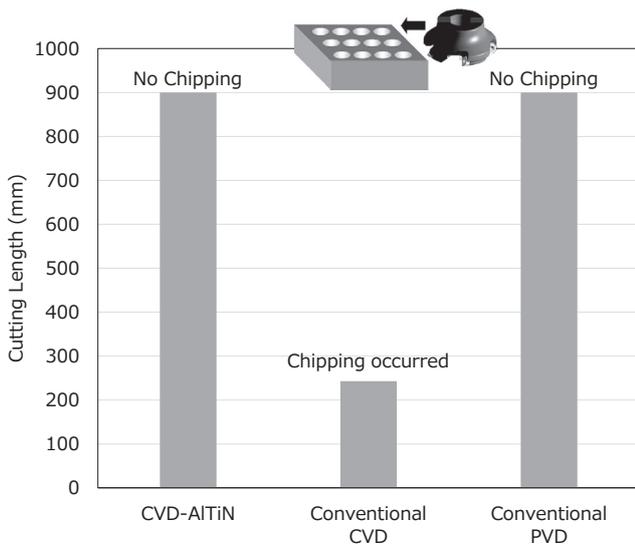
Figure 6 illustrates the chipping resistance evaluation results for CVD-AlTiN. Using S50C blocks with many holes in them as workpieces, an intensely interrupted face milling

was conducted. The edges were subjected to high loading by means of repeated contact impacts and their resistance to chipping was evaluated based on the cutting length before chipping. The cutting conditions used for the evaluation were: $V_c = 200$ m/min, $f_z = 0.50$ mm/t, $a_e = 60$ mm, $a_p = 2.0$ mm, and Dry. As in the case of wear resistance evaluation, the conventional CVD and PVD were also evaluated on the same occasion for comparison purposes. In this evaluation, the conventional CVD exhibited chipping at a cutting length of 250 mm. In contrast, both conventional PVD and CVD-AlTiN exhibited no chipping up to a cutting length of 900 mm. Thus, CVD-AlTiN proved itself to be resistant to chipping comparably to the conventional PVD.



Work: JIS:FCD700 Block
Cutter: WGC3160, insert: SEET13T3AGSNG, substrate: K20 grade
 $V_c=150$ m/min, $f_z=0.20$ mm/t, $a_e=80$, $a_p=2.0$ mm, Dry

Fig. 5. Wear resistance evaluation results

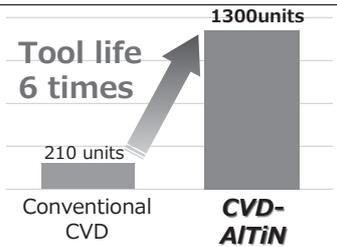


Work: JIS:S50C Block (Heavy interrupted)
Cutter: WGC3160, insert: SEET13T3AGSNG, substrate: P30 grade
 $V_c=200$ m/min, $f_z=0.50$ mm/t, $a_e=160$, $a_p=2.0$ mm, Dry

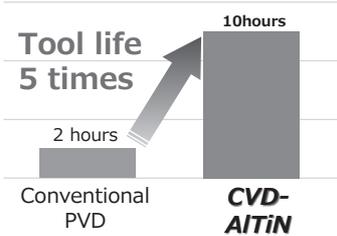
Fig. 6. Chipping resistance evaluation results

Figure 7 shows an example of actual machining use of CVD-AlTiN by a customer. Both examples (a) and (b) involved the machining of engine parts made of cast iron (FC250). While the machining described in example (a) conventionally used a CVD grade, sporadic chipping made the tool life unstable and the machining was limited to a specified capacity of 210 units. The use of a CVD-AlTiN tool for this process resulted in reduced chipping, enabling stable cutting up to 1,300 units, improving to 6 times that of the conventional CVD. While the machining described

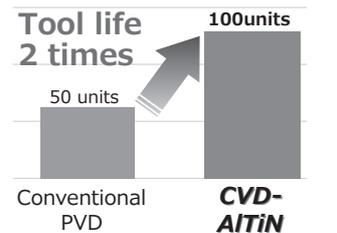
(a) Cylinder block (FC250)
 $v_c=300$ m/min, $f_z=0.26$ mm/t, Dry
WEX/AXMT170508PEER-G K



(b) Cylinder block (FC250)
 $v_c=300$ m/min, $f_z=0.26$ mm/t, Dry
WEX/AXMT170508PEER-G K



(c) Chassis parts (boron steel)
 $v_c=332$ m/min, $f_z=0.1$ mm/t, Dry
WGX/SEMT13T3AGSR-G P



(d) Hydraulic parts
(FC250/FCD450/SC450 mixed line)
 $v_c=236$ m/min, $f_z=0.12$ mm/t, Dry
DFC/XNMU060604PNER-G P K

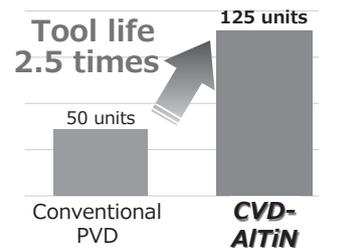


Fig. 7. Customer Evaluation results

in example (b) adopted a PVD grade with the emphasis on chipping resistance, the use of a CVD-AlTiN tool for this process made the machining as stable as the conventional PVD. Additionally, the tool's superb wear resistance made it possible to increase the number of machined units, enabling continual machining for 10 h, an improvement to five times that of the conventional PVD. Machining example (c) involved automotive chassis parts made of boron-alloyed steel. This process conventionally used a PVD grade emphasizing stability. However, the use of a CVD-AlTiN tool made stable machining possible at a doubled tool constant due to improved wear resistance. Example (d) presents the use of CVD-AlTiN in a line that handled cast steel and cast iron in a mixed manner. Because of the necessity for stable cutting of cast iron and cast steel differing in machinability, this process also conventionally used the conventional PVD. In example (d), the CVD-AlTiN tool with high levels of both chipping and wear resistance enabled the tool life to improve to 2.5 times that of the conventional PVD.

4. Conclusion

Using a nanometer-scale structural control technology based on CVD, Sumitomo Electric deposited a high-Al-content $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ coating and had success in applying it as Nano-Lamellae CVD-AlTiN on a mass-production basis. This new coating technology goes beyond the conventional CVD and PVD coatings. Application of this technology, named ABSOTECH X, will begin with the new milling grades XUC2500 and XCK2000 to be launched in 2021. Use of tools drawing on this technology will enable machining to be performed at a substantially higher speed and higher efficiency than for conventional machining and is likely to reduce machining costs at the customer end through improved cutting efficiency and extended tool life.

• ABSOTECH is a trademark or registered trademark of Sumitomo Electric Industries, Ltd.

Technical Terms

- *1 Cemented carbide: A composite material made from ceramics and metal, with its chief components being tungsten carbide (WC) and cobalt (Co).
- *2 Transverse rupture strength (TRS): An indicator of bending strength determined by a three-point bending test; Test method: CIS 026 (JIS R 1601, ISO 3327).
- *3 Spinodal decomposition: Phase separation corresponding to the change in state from an unstable state to equilibrium.
- *4 Shot peening treatment: A treatment intended for work hardening and imparting residual compressive stress through plastic deformation, by striking workpiece surfaces with many fine round particles at high speed.

References

- (1) Okuno S, et al., New Coated Carbide Grades for Cast Iron Turning, SEI TECHNICAL REVIEW, Vol.85 PP.44-47 (2017)
- (2) Leyendecker T, et al., The development of the PVD coating TiAlN as a commercial coating for cutting tools, Surf Coat Technol. vol.48, pp.175-178 (1991)
- (3) Paseuth A, et al., Deposition and analysis of Al-rich $c\text{-Al}_x\text{Ti}_{1-x}\text{N}$ coating with preferred orientation, J Am Ceram Soc vol. 100, pp. 343-353 (2017)

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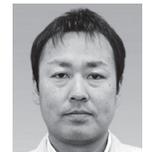
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