

Deposition of Ti–C–N coatings onto large-diameter tip saws

M. Tamura*, T. Tokunaga

Advanced Technology Research Laboratories, Nippon Steel Corporation, 3-35-1 Ida, Nakahara, Kawasaki, Kanagawa 211-0035, Japan

Abstract

Some steel pipes can be smoothly cut by using a saw with a tip of diameter of over 0.5 m. But martensite stainless steel is hardly cut by a standard-tip saw because of its hardness. Ti–C–N hard coatings were applied to a tip saw and the cutting performance of the coated tip saw was characterized. For many years hard coatings have been applied for many kinds of cutting tools. But there are some difficulties applying the hard coatings onto large-diameter-tip saws. The temperature of the substrate should be kept at under 250°C to avoid heat distortion of the tip saw. The same coating performance, such as hardness, thickness or adhesion is needed for each tip. Control of composition, thickness, and adhesion of coatings deposited at low substrate temperatures were studied by using reactive ion plating activated by arc-discharged plasma. Ionization of the reactive gas and ion-impact at an early stage of deposition were effective in improving adhesion. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Ti–C–N coatings; Tip saw; Cutting performance; Ion plating

1. Introduction

Using tungsten carbide-tipped saws for cutting steel pipes is effective in obtaining a smooth cutting section, which reduces odd portions during the manufacturing process and increases total production rate in steel works. In addition, heat damage to the cut materials is limited compared to the high-power cutting methods such as laser or torch cutting. Some steel pipes are smoothly cut by using a tip saw with a diameter of over 0.5 m. It is known that a large-diameter saw can reduce the resistance against cutting and show high cutting performance.

The increasing use of stainless steel pipes is accompanied by ever stricter demands on their mechanical and chemical properties. In order to meet these requirements, martensite stainless steel pipes have been developed. But martensite stainless steel is hardly cut by a standard-tip saw because of its hardness of HV500. It is necessary to pre-soften martensite stainless steel by heat treatment (which is a costly and difficult process) in order to cut it using a standard-tip saw. The goal of this study was to develop a high-performance tip saw which can be used for martensite stainless steels without any softening treatments. Ti–C–N hard coatings were applied to the tip saw, and the cutting performance of the coated tip saw was characterized.

For many years hard coatings have been applied for many kinds of cutting tools. Also, industrial applications of

wear protective multicomponent and multilayer coatings for cutting tools have been proposed in various processes [1]. But there are some difficulties applying the hard coatings onto large-diameter-tip saws. The temperature of the substrate should be kept under 250°C to avoid heat distortion. The same coating performance, such as hardness, thickness and adhesion, is needed for each tip. Control of composition, thickness, and adhesion of coatings deposited at low substrate temperature was studied by using ion plating, and the effect of the ions during deposition on the physical properties of the coatings was determined.

2. Experimental details

2.1. Deposition process

For the deposition of the coatings, reactive ion plating with an electron beam evaporator was used. The schematic of the experimental set-up is shown in Fig. 1. The substrate was sputter etched before deposition using an argon pressure of 25 Pa, a current of 0.2 A and a voltage of 500 V for a period of 30 min. Titanium of 99.9% purity was evaporated by using an electron beam with a power of 5 kW. The titanium vapor was activated and ionized by an ionization electrode. A current of 4.0 A was obtained through the ionization electrode. The coatings were deposited on the negatively biased substrates. A voltage of 300 V as a negative bias was applied on the substrates and tip saw during deposition. The effect of ion impact at the

*Corresponding author. Tel.: +81-44-7971244; fax: +81-44-7525759; e-mail: tamura@lab1.nsc.co.jp

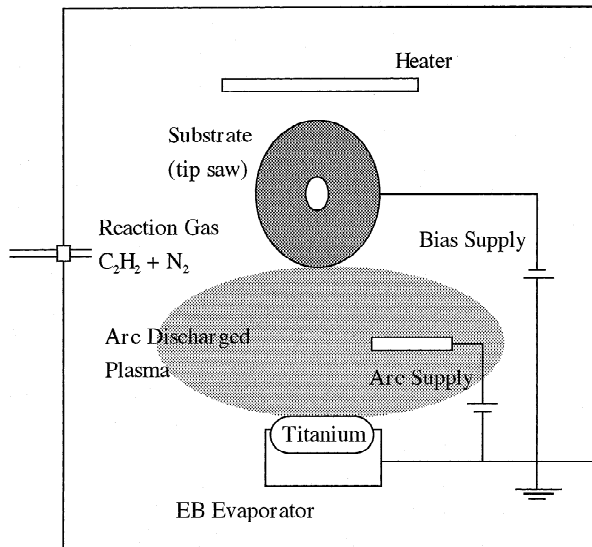


Fig. 1. Schematic diagram of the ion plating system used in this study.

early stages of deposition was studied by applying a negative bias of 400 V for a period of the first minute of deposition. The coatings were deposited on the substrates in a $C_2H_2-N_2$ gas mixture. The total pressure was held at 0.05 Pa and the deposition time was 60 min. The deposition rate was $0.1 \mu\text{m}/\text{min}$, giving a total thickness of 6 μm .

The TiN coatings were deposited onto tungsten carbide to study fundamental properties of the coatings, such as adhesion, microstructure and thickness distribution at low substrate temperature. In the previous study [2], there was very little effect of composition of the Ti–C–N coatings on microstructure. The adhesion results for TiN can be applied to the Ti–C–N coatings as well. Sufficient adhesion is necessary for both TiN and the Ti–C–N coatings deposited at low substrate temperatures.

It has been reported that the Ti–C–N coatings showed more resistance against wear than TiN coatings [2]. Ti–C–N coatings of TiN (2 μm), $TiC_{0.6}N_{0.4}$ (3 μm) and TiN (1 μm) were applied onto the tip saw for the cutting tests. The coatings were deposited at a substrate temperature below 250°C . The tip saw was rotated above the EB evaporator at 2–3 rpm.

2.2. Characterization of the coatings

Fundamental properties of the coatings were characterized by X-ray diffraction analysis (XRD), glow discharge spectroscopy (GDS), electron-probe microanalysis (EPMA), and scanning electron microscopy (SEM). GDS was used to study the chemical composition depth profile of the Ti–C–N coatings. EPMA was carried out to analyze the chemical composition of the coatings with a calibration using TiC and TiN standard ceramics. The hardness was measured on the as-deposited coating surface with a

Vickers microhardness tester under a load of 50 gf with a dwell time of 15 s. At least five indentations were measured for each sample. The structure and the size of grains in the coatings were studied using SEM and transmission electron microscopy (TEM). XRD was performed to analyze the phases in the coatings. The residual stress in a coating on a disk substrate can be determined by the difference in deflection of the disk before and after coating deposition [3,4]. The internal stress of the coating can be calculated by this method without knowledge of the elastic constants of the coating. The adhesion of the coatings was tested by scratch testing on a CSEM Revetest (Centre Suisse d'Electronique et de Microtechnique, Neuchatel). The diamond was a Rockwell C type with a tip radius of 200 μm . Samples were tested under a continuously increasing load on the diamond with a horizontal speed of 10 mm min^{-1} .

2.3. Cutting test

Two kinds of cutting tests were performed. They are shown in Table 1. Condition 1 corresponds to manufacturing of carbon steel pipes whose tensile strength is 0.49–0.71 GPa (50–72 kg/mm^2). Carbon steel pipes whose outer diameter was 340 mm and thickness was 10 mm were cut by a tungsten carbide-tipped saw under dry conditions. A schematic of a tungsten carbide-tipped saw is shown in Fig. 2a. Tungsten carbide chips are attached to a round carbon steel plate. Cutting performance was evaluated by total cutting area of steel pipes until the flank wear of the tungsten carbide tips exceeded 1.5 mm.

Condition 2 corresponds to the cutting of martensite stainless steels whose tensile strength is 1.47 GPa (150 kg/mm^2). Martensite stainless steel rods with a rectangular cross-section of area 0.49 m^2 were prepared for the cutting tests. A large-tip saw whose diameter was 810 mm was coated and used for cutting hard steels as shown above. After one or two cuts ($0.49\text{--}0.98 \text{ m}^2$) of the martensite stainless steel rods, a tip saw without a coating cannot be

Table 1
Conditions of cutting tests

	Condition no.	
	1	2
Work	Carbon steel	Stainless steel
Tensile (GPa)	0.49–0.71	1.047
Hardness (Hv)	200–300	500
Shape	Pipe	Rods
Section (m^2)	0.01	0.49
Saw		
No. of tips	50	48
Diameter (mm)	340	810
Thickness (mm)	3.7	7.5
Cutting		
Speed (m min^{-1})	200	100
f (mm)	0.06	0.07
Condition	Dry	Using oil

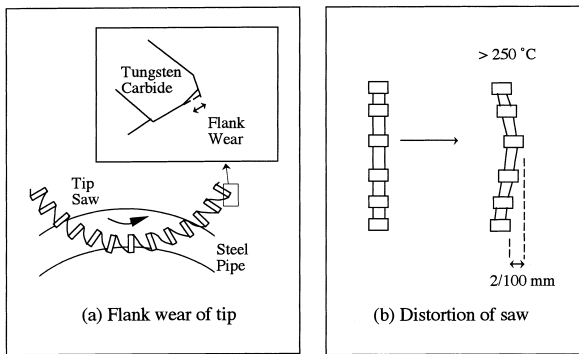


Fig. 2. Tungsten carbide-tipped saw.

used further because the attached tips are very worn and have chemically reacted with the working steels. After seven cuts (3.4 m^2) using a Ti–C–N-coated tip saw, the adhesive and wear performance of the coatings of the tungsten carbide chips were observed using a microscope.

Tungsten carbide-tipped saws are very sensitive to heat treatment. Distortion (Fig. 2b) of over 0.02 mm should be avoided to obtain a sufficient cutting performance of the tip saw. Distortion of 0.05 mm can be caused by a heat treatment at a temperature of over 400°C . Distortion of 0.02 mm was measured by annealing the tip saw at 250°C . This means that the coating should be deposited below a substrate temperature of 250°C .

3. Results and discussion

3.1. Adhesion

At a low substrate temperature during deposition, sufficient adhesion between the coating and the substrate is not obtained. The critical load (L_c) in the scratch test is greater than 20 N for commercially coated cutting tools. At a substrate temperature of 500°C , the L_c of a TiN coating deposited by the ion plating system in this study was 25–35 N, which is sufficient adhesion for cutting tools. But, at a substrate temperature of 200°C , the L_c was 10–14 N, which is not sufficient. In order to improve the adhesion of the coatings deposited at low substrate temperatures, relaxation of residual stress in the coatings and increasing of bonding strength of the interface were studied.

Peeling of the TiN coating from the substrate was observed after 24 h of deposition as shown in Fig. 3. This coating was deposited on the substrate at room temperature. Cracks initiated from some impurities or defects and extended in a circular pattern as time passed. This implies there is high residual stress in the coating.

Compressive residual stresses in the TiN coatings were calculated from 2.05 to 3.65 GPa as shown in Fig. 4. Observed values of residual stress in the TiN coating in this study were consistent with published values. Published values of residual stress in TiN coatings vary from 0.5 [5]

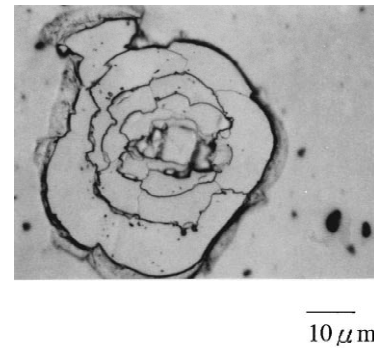


Fig. 3. Self-peeling of TiN deposited at room temperature.

to 5.0 GPa [6], and largely depend on deposition process and conditions. The substrate temperature during deposition would be an important factor for residual stress in the coatings. Fig. 4 shows the effect of substrate temperature during deposition on residual stress in the TiN coating. Higher residual stress (3.00–3.65 GPa) was observed in the coatings deposited at a substrate temperature of 200°C than that (2.05–2.75 GPa) in the coatings deposited at 500°C . The residual stress of the coating consisted of intrinsic stress resulting from the growth process and thermal stress arising from the thermal expansion mismatch between the coating and the substrate cooling from the deposition temperature. Although it is difficult to know the value of intrinsic stress in the coatings directly, the thermal stress can be easily calculated from thermal expansion coefficients of the coating and of the substrate [7]. The thermal stress in the TiN coating deposited on tungsten carbide at 200°C was calculated as 0.8 GPa. This implies that the residual stress in the TiN coating at low substrate temperature is mainly affected by intrinsic stress in deposition. The intrinsic stress was the result of energetic ions and neutral bombardment during deposition [8].

Relaxation of high residual stress in the coating may improve adhesion of the coating deposited at low substrate temperature. Heat treatment can affect the residual stress in

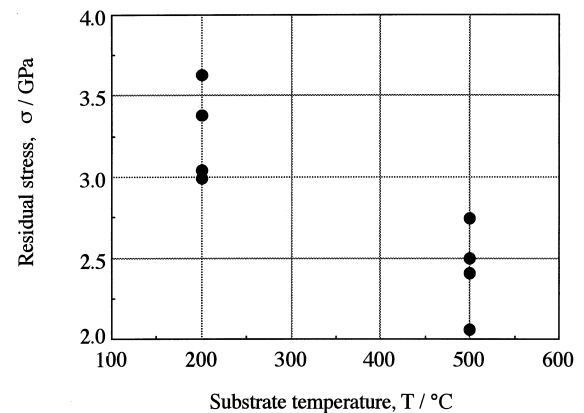


Fig. 4. Effect of substrate temperature on residual stress.

the coatings and annealing effects can be described in terms of the recovery of interstitial atoms introduced during deposition [9]. The hardness–temperature response implies a relaxation of residual stress in the coatings. Fig. 5 shows the hardness hysteresis of a Ti–C–N coating. It was observed that the hardness values recorded on cooling from 900°C were less than those on heating. The residual stresses of the coatings were found to decrease to nearly zero by annealing above a temperature of 900°C. The relaxation occurs only by annealing above a temperature of 900°C which is too high for the heat treatment of the tip saw and is not applicable.

The effect of ion impact on adhesion was studied. Higher bias (–400 V) for a period of 1 min at early stage of deposition was applied to the substrates. A bias voltage of 400 V cannot be applied for more than 1 min because the ion impact of this caused a rapid increase of substrate temperature. The results are shown in Fig. 6. Applying higher bias at an early stage of deposition was very effective for improving adhesion. A L_c value of 28 N, which was applicable for cutting tools, was obtained even at a substrate temperature of 200°C during deposition. TEM analysis (Fig. 7) of this adhesion-improved sample shows there is a dense defect zone at the interface between the TiN and tungsten carbide substrate. The dense defect zone whose thickness was 0.05 μm could be formed by ion impact.

Fig. 8 shows the effect of ion bombardment during deposition on grain size and texture of the coating. Fine-grained and dense coatings were formed by the bombardment of ions during deposition. The grain size of the TiN coating obtained by vacuum evaporation was 0.1–0.2 μm which was much larger than the grains (0.01–0.05 μm) in

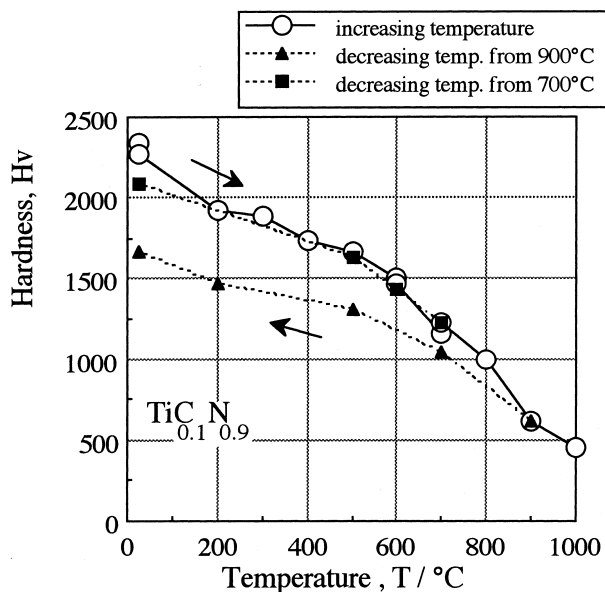


Fig. 5. Hv hysteresis of the Ti–C–N coating. Hv was measured in vacuum at each temperature.

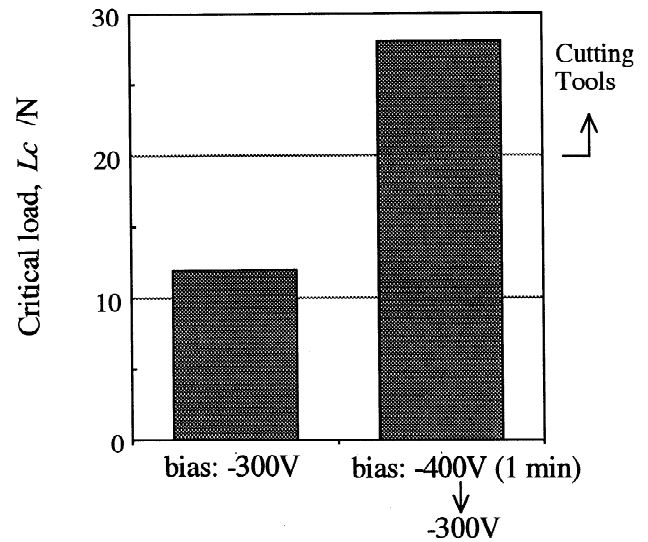


Fig. 6. Effect of bias applied at an early stage of deposition on the critical load. The TiN coating was deposited at 200°C.

the coatings deposited by ion plating. Fine-grained coatings could be formed from dense nucleation sites induced by ion impact on the surface of the substrate. It is concluded that the ion-impact affected zone could enhance the nucleation of TiN and, as a result of this, the interfacial bonding strength could be improved.

The structural changes in the materials are known after ion bombardment. The ion bombardment leads to an increase of up to several-fold in the density of etching pits in comparison with the initial density, at a distance from the surface of several μm . A noticeable increase in the density of defects, such as micropores, dislocations, dislocation loops and their accumulation, was observed on the surface [10].

The grain size is of overwhelming importance in affecting the strength of polycrystalline materials, and hard

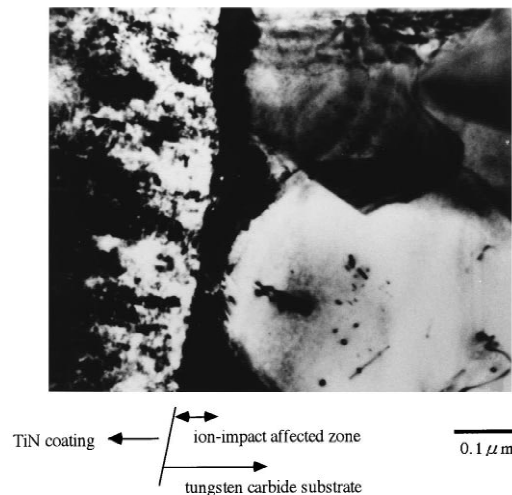


Fig. 7. TEM analysis of the interface between the TiN coating and tungsten carbide.

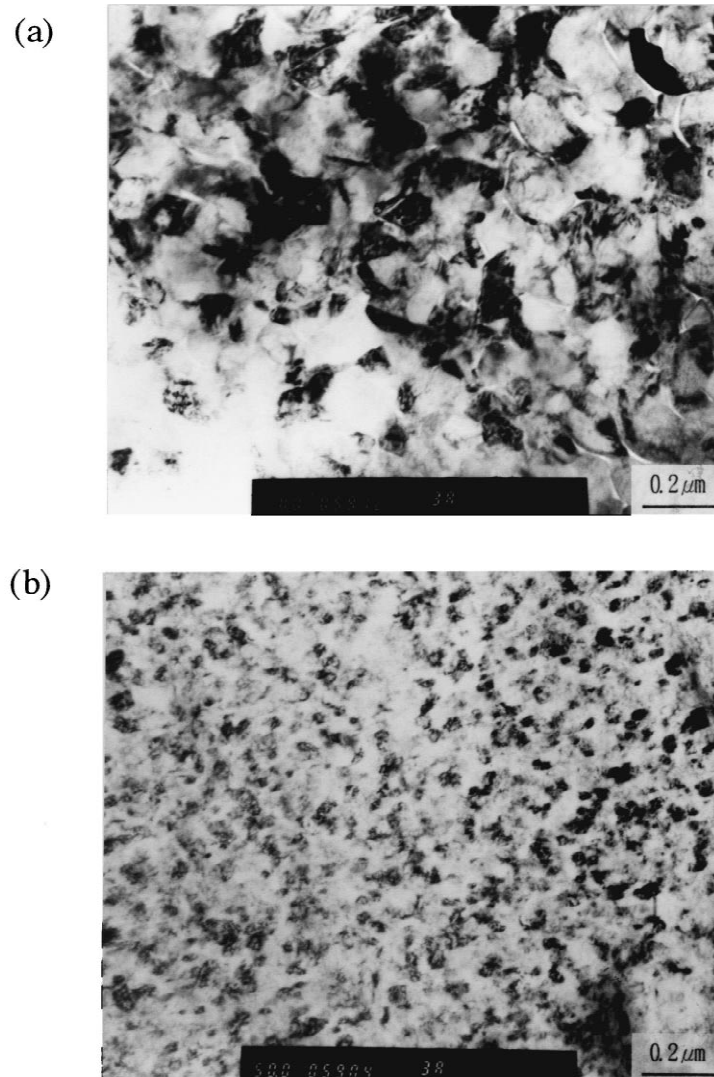


Fig. 8. TEM micrograph of the TiN coating. EB power, 5 kW; substrate temperature, 300°C; P_{N_2} , 0.05 Pa. (a) Vacuum evaporation, (b) ion plating (substrate current, 3.89 A m⁻²).

coatings are no exception. A Hall–Petch relationship has been confirmed in PVD metal coatings [11]. The fine-grained coating deposited by ion plating shows a high value of hardness and this is suitable for wear-resistant materials such as cutting tools.

3.2. Thickness distribution

For deposition of coatings for large-diameter substrates, the difference in deposition rate at each position should be minimal. In other words, the thickness distribution should be dependent only on the distance between substrate and ion source. This is necessary in order to obtain uniform properties of coatings for large substrates. Especially for tip saws, identical coating performance, such as hardness, thickness and adhesion, is needed for each tip. This is important because one worn tip directly affects the lifetime of the tip saw.

For vacuum evaporation, the maximum deposition rate was obtained just above the ion source, as shown in Fig. 9. The TiN coating was deposited at a constant distance from the evaporation source and substrate, and at constant evaporated weight of 6.0 g titanium. Ionization power is shown beside the figure. Ionization of 0 V–0 A corresponds to vacuum evaporation. A thick coating deposited by vacuum evaporation was porous and a dense coating was obtained by ion plating. This is consistent with TEM observations, as shown in Fig. 8. Ionization was effective for uniform thickness distribution at the constant substrate-to-ion source distance.

3.3. Cutting performance

Results of cutting performance at condition 1 are shown in Fig. 10. Deposition of coating while maintaining the substrate temperature under 250°C was successful. After

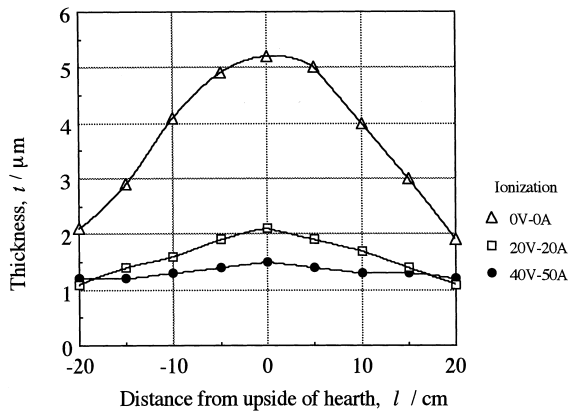


Fig. 9. Effect of ionization on thickness distribution. TiN was deposited at a constant distance of 40 cm from the EB source. Weight of evaporated Ti was 6.0 g.

the deposition, the distortion of the tip saw was within 0.02 mm. Fig. 10 shows that the cut area of work steel pipes could be extended 4-fold by applying Ti–C–N coating.

A Ti–C–N-coated tip saw before cutting under condition 2 is shown in Fig. 11. After cutting 3.4 m² of hard steel, very little wear of the Ti–C–N coating was observed, as shown in Fig. 12. This means that ion-plated Ti–C–N coatings deposited at low substrate temperature by applying high bias at an early stage of the deposition was very effective in improving cutting performance of large-diameter tip saws.

4. Conclusions

Ti–C–N hard coatings were applied on tip saws. At low substrate temperature during deposition, sufficient adhesion between the coating and the substrate is not obtained.

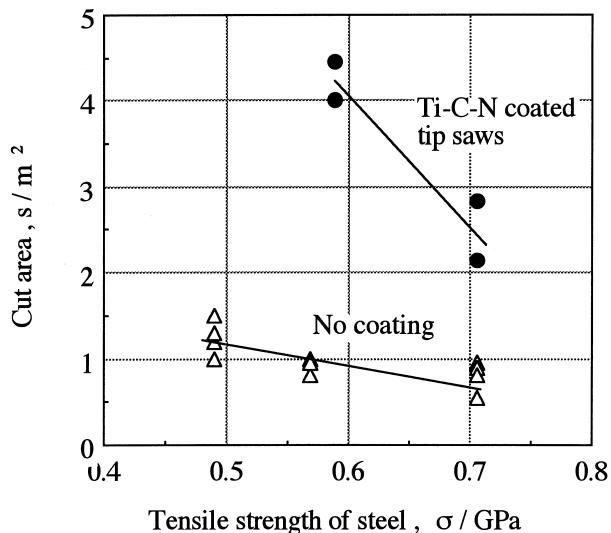


Fig. 10. Cutting performance of Ti–C–N-coated tip saws. Carbon steel pipes were cut. Diameter of saws, 280 mm.

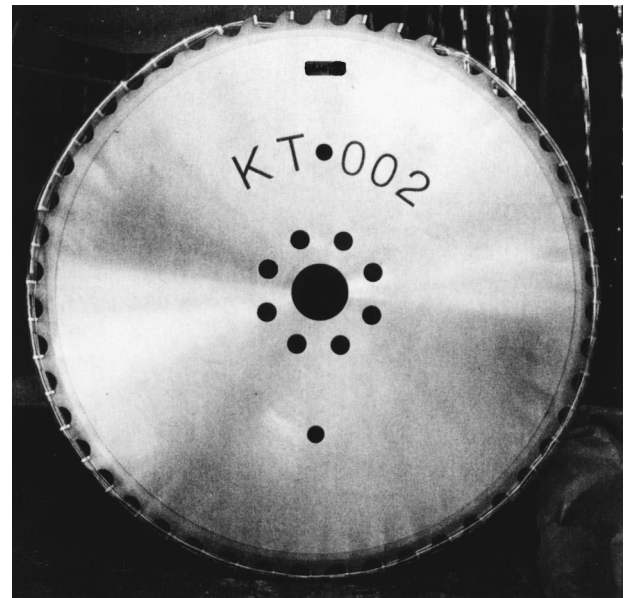


Fig. 11. Ti–C–N-coated tip saw used for cutting of hard steel (diameter of saw, 810 mm).

The substrate temperature during deposition is an important factor for residual stress in the coatings. Higher residual stress was observed in the coatings deposited at lower substrate temperature. But the relaxation of the residual stress occurs only by annealing over a temperature of 900°C, which is too high for the heat treatment of the tip saw and is not applicable. Applying higher bias at an early stage of the deposition was very effective in improving adhesion. TEM analysis of this adhesion-improved sample shows there is a dense defect zone at the interface between the TiN and tungsten carbide substrate. A dense defect zone of thickness 0.05 μm could be formed by ion impact. The ion impact-affected zone could enhance nucleation of the TiN and, as a result, the bonding strength of the interface could be improved. Ionization during deposition



Fig. 12. Ti–C–N-coated tips after cutting 3.4 m² of hard steel, showing good adhesion and wear resistance of the coating.

was also effective for uniform thickness distribution at a constant substrate-to-ion source distance. This is helpful, especially for large-diameter tip saws whose tips should have the same performance. Ti–C–N-coated large-diameter tip saws showed excellent cutting performance for carbon steel pipes and martensite stainless steels.

References

- [1] O. Knotek, F. Löffler, G. Krämer, *Surf. Coat. Technol.* 54–55 (1992) 241–248.
- [2] M. Tamura, K. Fukuda, *ISIJ Int.* 33 (1993) 949–956.
- [3] A. Pan, J.E. Greene, *Thin Solid Films* 78 (1981) 25.
- [4] J.A. Sue, *Surf. Coat. Technol.* 54–55 (1992) 154–159.
- [5] D.S. Rickerby, B.A. Bellamy, A.M. Jones, *Surf. Eng.* 3 (1987) 138.
- [6] A. Kinbara, S. Baba, N. Matuda, *Thin Solid Films* 141 (1986) 229.
- [7] G. Kleer, R. Kassner, E.-M. Meyer, M.G. Schrinker, W. Doell, *Surf. Coat. Technol.* 54–55 (1992) 167–172.
- [8] T.J. Vink, J.B.A.D. van Zon, *J. Vac. Sci. Technol.* A9(1) (1991) 124.
- [9] H. Suzuki, H. Matsubara, A. Matsuo, K. Shibuki, *J. Jpn. Inst. Met.* 49 (1985) 773.
- [10] G. Kuznetsov, V. Delian, *Surf. Coat. Technol.* 54–55 (1992) 96–101.
- [11] J.A. Augis, C.C. Lo, M.R. Pinnel, *Thin Solid Films* 58 (1979) 457.