Wear Behaviors of Ceramics TIN, TIC and TICN with Arc Ion Plating

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In order to determine the wear properties of AIP(Arc Ion Plating) deposition, wear process was evaluated by using a Falex test machine. Also, in order to determine the effects of coating material on the wear process, TiC, TiN, and TiCN coatings of thickness about 5 μm~6 μm coated by Arc ion plating deposition method were tested. The wear property was determined under a dry sliding condition as a function of the applied load, sliding distance, sliding velocity and temperature. The results show that when wear of the coating-layer occurred, specific wear amount increased with the wear rate. At initial state, the wear rate rapidly increased, but it gradually reduced as the velocity increased. Also, when raising the temperature, the wear rate increased in the order of TiCN, TiN and TiC due to the frictional heat.

Key Words: Friction, Wear, Tribology, Arc Ion plating, PVD, Specific Wear Rate

1. Introduction

Along with the development in the industry, the mechanical components used in various industries require stronger materials to endure severer environments. As the overall mechanical industry rapidly develops and the variety of machines become more specialized and precise, the working conditions require higher speeds and heavier loads. Thus, solutions to tolerate the surface conditions of all composition of the machine are necessary. Therefore, many researches have been conducted to improve the characteristics of the metal surface (Song and Ahn, 1990; Ensinger and Schröer, 1998).

Coating, a kind of surface treatment, was often used to protect the substrate beneath the coating from outside dangers at the beginning. However, recently it has taken an important role as an integral part of the machine with many characteristics that are required by forming the coating film as well as the original purpose of protecting the substrate.

Coatings include CVD(Chemical Vapour Deposition) and PVD(Physical Vapour Deposition). CVD includes MCVD(Modified Chemical Vapour Deposition) (Cho and Choi, 1994; Hocking, 1989) and Plasma Spraying, which are high heating sources and suitable for spraying of ceramic materials with high melting point. Plasma spraying is applied to production of aircraft related components.

PVD can be usually divided into Vacuum Deposition, Sputtering and Ion Plating. One of Ion Plating methods, AIP(Arc Ion Plating) (Vyskocil and Musil, 1990; Tai and Koh, 1990; Peterson and Winer, 1980) is widely used in coating of wear resistant mechanical components (Honlmberg and Matthews, 1994; Halling, 1983;

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Lee and Kim, 1994). AIP of a film grown from the vapour phase with a beam of energetic ions is a coating technique which has been studied for a number of years and is presently being developed for industrial applications (Smidt, 1990; Hirvonen, 1991; Hubler, 1994; Cuomo and Rossanagel, 1989). Particularly compound films, such as metal oxides for optical purposes and nitrides and carbides for wear protection, look promising, owing to their special features, such as low microporosity and high hardness and adhesion (Cuomo et al., 1989). This technique can be used to deposit oxides, nitrides, and carbides of titanium (Barth and Ensinger, 1990; Schroer and Ensinger, 1996). Also, Titanium has many excellent features such as high strength-to-weight ratio, good high-temperature properties, and excellent corrosion resistance and biocompatibility (Garside, 1991; Ruck et al., 1995, Sioshansi and Oliver, 1985, Dearnaley, 1978; Oliver, 1984).

Unfortunately, despite of such superior wear resistance of ceramics, the study of ceramic friction and wear shows big variations in the coefficient of friction and wear rate depending on surface type and experimental conditions (Lee and Son, 1996; Hisakado, 1992). Also, as most of the studies have been carried out in specific fields only, there were few extensive studies on the wear of ceramic materials (Anderson, 1992; Rhee, 1995; Halling, 1983).

In this work, we investigated the efficacy of three coafings, TiN, TiC and TiCN, by measuring the specific wear rate with respect to the load, temperature and sliding velocity.

2. Experiment

2.1 Experimental devices

For the experiment, a Falex test machine was used after specimen surface preparation to study the wear characteristics as a function of load, speed, temperature and time changes. Figure 1 shows a schematic diagram of the test machine and Fig. 2 describes the enlarged V-blocks and journal arrangement.

Testing part consisted of the V-blocks and the journal. The journal is in contact with two V-blocks under the applied load. Standard test cylinder type of the journal is 6.35 mm in outer diameter, thermal treated AISI 3135 steel with 31.75 mm in length and 87-91 in hardness. For the two V-blocks, thermal treated AISI 4130 of 20-40 in hardness was used. The angle of the V-blocks layers was 96±1°. Coating was done only on the V-block. Journal can slide and rotate between the two blocks. Table 1 shows the physical and chemical properties of the journal and the V-blocks.

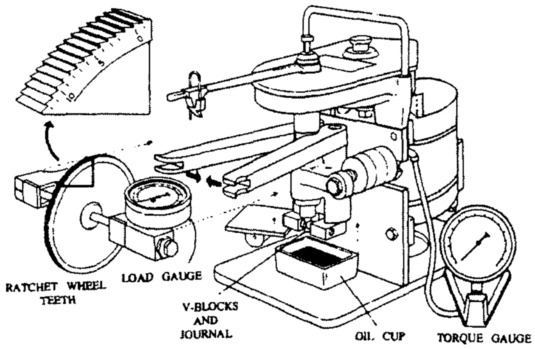


Fig. 1 Schematic diagram of test machine

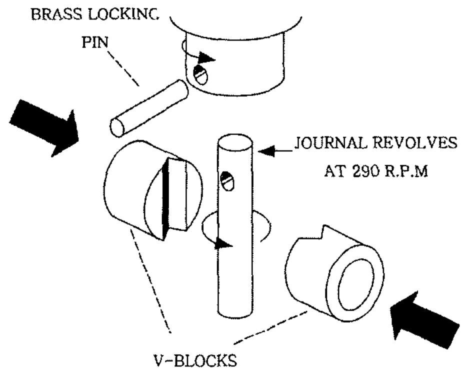


Fig. 2 Exploded view of V-block and journal arrangement

The load action part was made of a ratchet wheel system. The load could be delivered to the V-blocks through a lever arm spring gauge.

2.2 Creation of solid lubricat film

For ceramic coating, an arc ion plating equipment was used. Coating was made in the following order. The chemically cleaned tools were inserted in the furnace. The temperature was increased to the desired temperature (℃) by using a preliminary heating source. Also, the chamber was evacuated using a vacuum pump. Then ion cleaning, titanium coating and the main coating were performed.

In the ion cleaning process, the titanium target was triggered to generate titanium ions and a negative voltage of about 500V or more was applied to the substrate under a high vacuum of 5×10^{-5} Torr. Through this process, Ti ions became accelerated and collided with the substrate to remove the foreign materials and the oxidation layer. The voltage was lowered to less than 400V for Ti middle layer coating and Ti ions were coated on the surface of the substrate. At this stage, the thickness of the titanium middle layer was adjusted to 0.1-0.3 μm. This process was performed to improve the adherence between the coating layer and the substrate. Then, by lowering the negative voltage (V) and inserting a reactive gas (CH, N) of given amount at the main coating stage, the desired compound with high hardness could be formed on the surface of the substrate. It was found that high temperature was more advantageous unless it caused transformation or softening of the substrate. This is because the higher the temperature, the movement of the chemical species that participate in the reaction on the surface of the substrate become easier during coating. In addition, high temperature makes a more dense structure.

In this study, the temperature in the furnace was kept between 500-700℃ to consider the softening temperature of the AISI 3135 steel substrate. When the substrate is fixed in the furnace or if it just rotates, there may be some difference in film the thickness depending on the location of the substrate. Therefore, the substrate was made to revolve and rotate at the same time to prevent such a problem.

3. Method of Experiment

3.1 Test on thickness and surface of coating film

To find out the thickness of the coating film and its surface status, we used a metallographic microscope. Also, to measure the thickness of the coating film, tapes were attached on some part of the surface before coating and removed to measure the thickness of the coating film using a SEM (Scanning Electron Microscope).

Table 2 shows the microstructural and mechanical properties of various materials.

3.2 Hardness test

As thin coating film was affected by the substrate depending on the load, the hardness was measured more than 10 times with a relatively small load, using a vickers microhardness tester and the average value was obtained.

3.3 Wear test

Figure 3 shows the overall process of TiC, TiN, and TiCN coating on the material surfaces.

Table 1 Physical and chemical properties of journal and V-block

Properties \ Items	Journal	V-block
Surface Roughness	$(1.3\times10^{-7}\sim 2.5\times10^{-7}$ m)	$(1.3\times10^{-7} \sim 2.5\times10^{-7}$ m)
Hardness	87～91[H_B]	20～24[H_{RC}]
Chemical Composition (wt%)	C : 0.15 Mn : 0.50 S : 0.04 P : 0.035 Si : 0.20 Cr : 0.35	C : 0.30 Mn : 0.45 S : 0.08～0.13 P : 0.04 Si : 0.20 Cr : 0.80

Table 2 Microstructural and mechanical properties of TiC, TiN and TiCN

Properties	TiC	TiN	TiC, N
Melting point, ℃	3160	2950	3300
Density, g/mm^2	4.90	5.2	5.78
Hardness Hv, kgf/mm^2	3200	2500	4000
Chemical stability	poor	good	very good
Frictioc coefficient with steel (dry condition)	0.76	0.65	0.55
Reactive Gas	CH_4	H_2	CH_4, N_2
Thermal expansion coefficient (10^{-5}/℃)	7.61	9.35	9.82

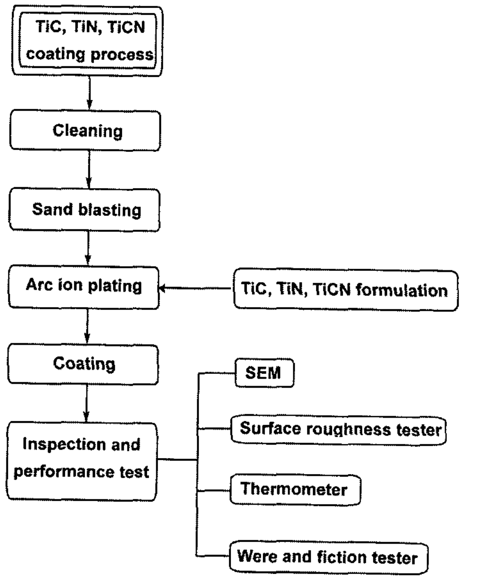


Fig. 3 Overall process for TiC, TiN, TiCN coating

Before coating the ceramic lubrication film on the substrate, the surface was cleaned using a non-polar solvent that does not create any film. After that, pretreatment was performed on the surface to improve the adherence between the film and the substrate. Pretreatment included polishing with 0.5 μm alumina particles, because the friction and wear can be affected by inconstant surface roughness. Also, sand blasting treatment was applied selectively so that the roughness becomes about 0.6–0.9 μm after treatment. The Falex friction and wear tester was used for friction and wear tests of the ceramic lubrication coating films.

The torque and load gauges were set to 0 and the load was changed by a ratchet wheel to 1112, 2223, 3334, 4445, 5556 [N]. The loads and pressures were measured by the gauges. All the experimental conditions and terms are presented in Table 3.

Figure 4 shows a wear model. Specific wear rate can be obtained with the following equation.

$$Ws=V/NS$$

where V denotes the wear volume. N and S denote the normal loads and the sliding distance, respectively. The test was performed 5–10 times under different conditions.

Table 3 Test condition of the experiment

Item	Test Condition
Sliding Speed (m/sec)	2.7
Applied load (KN)	1
Environment	Dry
Test Interval (min)	5–10
Temperatur Range (℃)	50–200

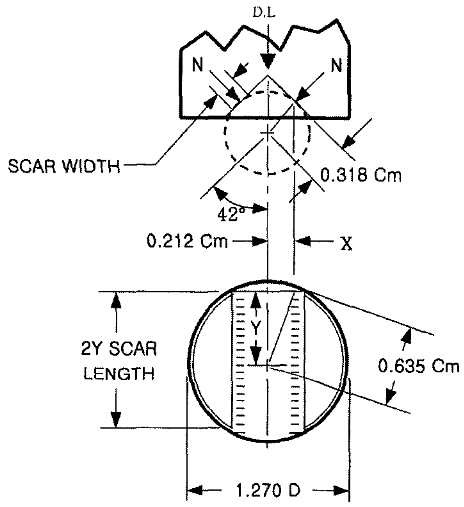


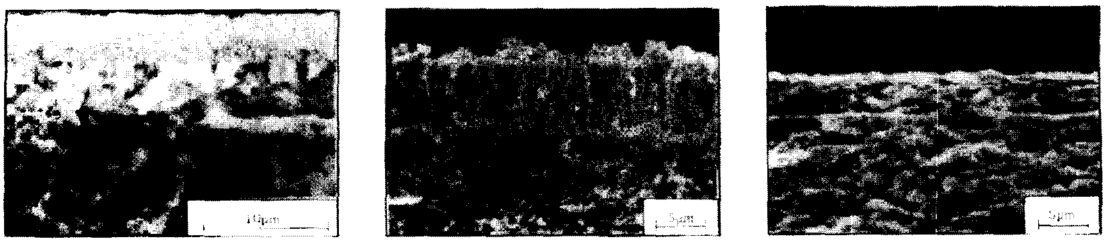
Fig. 4 Wear model for the contact of journal on V-block

The wear rate was obtained by calculating the average value after multiplying the number of cogs of ratchet wheel corresponding to the wear of 0.0254 mm by the wear scar area. Specific wear rate was calculated by dividing the wear rate by the load on the wear surface and the sliding distance.

4. Results and Discussion

4.1 Characteristics of coating layer

The surface of the coating layer reflects its crystallinity, crystal structure and structure of roughness, which are very important factors in ceramic coating. The thickness value of the coating film was 5–6 μm. The measureents were done using a SEM and a roughness tester, which indicated comparatively uniform results. As shown in Fig. 5, TiC coating film showed higher surface



(a) (b) (c)

Fig. 5 SEM micrographs for determination of coating film (a ; TiC b ; TiN c ; TiCN)

Table 4 Surface roughness and Hardness of Non-Coated Journal and Coated V-block

Specimen	Non-Coated Journal	TiC	TiN	TiCN
surface roughness, Ra (μm)	0.19	0.084	0.062	0.041
Hardness, Hv (Pa/mm^2)	200	2900	2300	3400

roughness value than TiN or TiCN coating films.

Table 4 shows the test result obtained by a portable roughness tester. Hardness test results also appear in Table 4. The hardness was obtained by average the value after measuring the hardness of the TiN coating film under 10g of load over 10 times. TiCN coating film showed higher hardness than TiC or TiN coating. Also, TiC had higher hardness than TiN and its crystal structure was more minute while the roughness was higher than TiN.

4.2 Wear characteristics as a function of load

Figure 6 shows the variation of wear rate as a function of load. In this figure, the wear rate is a result of measuring the wear width up to the 0.5 μm unit. As shown, the wear rate increases as the load increases regardless of the coating films. Under any load condition, TiCN coating showed rather less wear rate compared to TiC or TiN coatings. The reason is mainly due to the order of hardness ; TiCN > TiC > TiN. It can be stated that harder coating cause less wear rate. In addition, there was little increase in the wear rate as the load increased. When the load was increased from

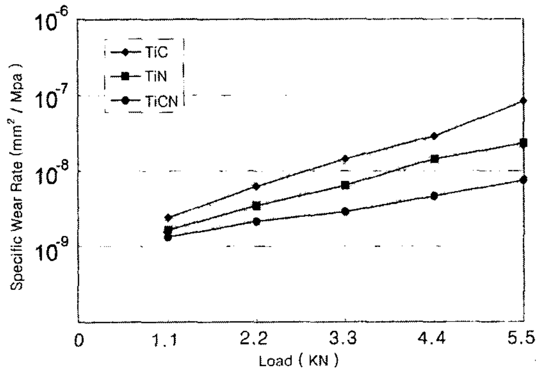


Fig. 6 The variation of specific wear rate as a function of applied load

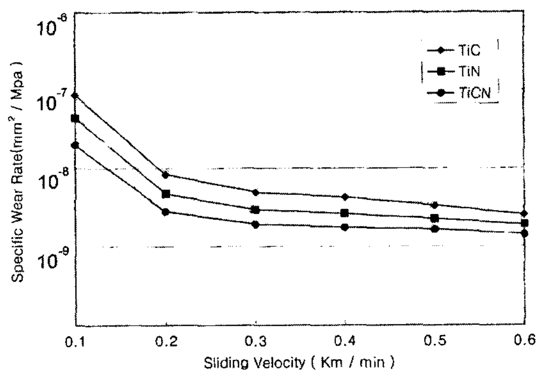


Fig. 7 The variation of specific wear rate as a function of applied sliding velocity

1112N to 5556N in 1112N increments, the wear rate was not increased significantly.

4.3 Wear characteristics as a function of velocity

Figure 7 shows the changes of specific wear rate as a function of velocity. As shown in the figure, TiN showed less wear rate than TiC. Also. TiCN had less wear rate than TiN in any velocity. All of the three coatings showed high wear rate at the initial area, but the specific wear rate was reduced as the velocity increased. We think that it was due to the frictional heat caused by increased velocity. The frictional heat could be transfered to the surfaces of TiC, TiN, and TiCN. This may cause the oxidized steel and film to form, preventing the wear of TiC, TiN, and TiCN.

In addition, the difference in the wear rate was reduced as the velocity increased.

4.4 Wear characteristics as a function of temperature

Figure 8 shows the specific wear rate as the temperature of the coated lubrication film changes. Wear reduced most significartly in TiCN and TiN followed by TiC. At the initial stage, the wear rate was slow, but rapidly increased from 150℃. The outer cover of each film had gradually been removed and the outside of each film was released as it passed the running-in effect at the early stage.

Besides, the contact surface was affected significqutly by the increased temperature with the frictional heat generated at the sliding contact as the temperature was increased by the external heat. Also, as the temperature of the contact surface was increased, the oxidation of the ceramic film was accelerated, and thus the lubrication film was destroyed. The difference in the wear rates between each substrate was increased by a similar amount.

4.5 Wear characteristics with different as a function of sliding distance

Figure 9 shows the changes of specific wear rate in respect to the sliding distance. The specific wear rate increased sharply until the distance reached 3240 m. The specific wear rate, however, increased slowly after 3240 m. As a result, the wear rate increased with sliding distance, but the increasing rate not prominent after some point. This is because the wear size becomes larger as wear progresses, and the effective stress experienced by the substrate is reduced even though the load is constant.

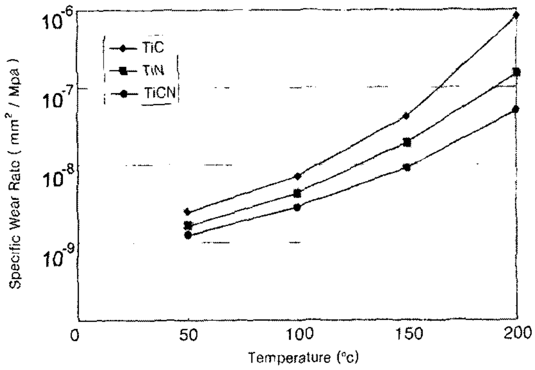


Fig. 8 The variation of specific wear rate as a function of applied temperature

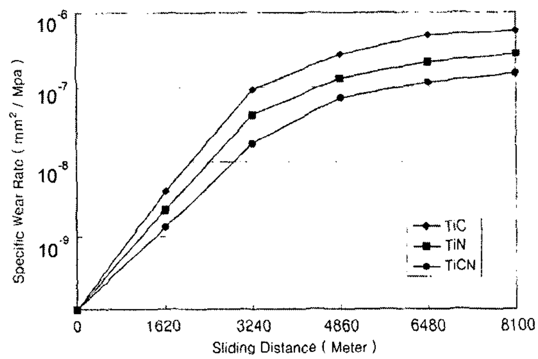


Fig. 9 The variation of specific wear rate as a function of sliding distance

There are some differences in the wear rates among TiC, TiN and TiCN with respect to the sliding distance. The reason is that TiC is easy to form cracks. Also once there is a crack, TiC which has high roughness can be peeled off more easily than TiN and TiCN.

5. Conclusion

We studied the wear properties of TiC, TiN and TiCN coatings on the V-block. The coating was done by using the arc ion plate process. wear tests were conducted using a Falex tester with respect to load, velocity and temperature.

The test results are as follows :

(1) As for the surface roughness, TiC is the roushest. TiCN has the highest hardness followed by TiC and TiN.

(2) Wear rate is increased with load, in the

order of comes TiC, TiN, and TiCN but the rate of increase is constant.

(3) At the initial stage, the wear rate rapidly increased, but it is gradually reduced with increased velocity. It becomes stable in the order of TiC, TiN and TiCN.

(4) With increasing temperature, wear is increased in the order of TiCN, TiN and TiC by the frictional heat. At first, the wear rate was slow, but was rapidly increased after 150℃. The contact surface is affected by the temperature.

(5) Wear rate is increased with sliding distance, but the rate of increase is not prominent after some point.

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References

Andersson, P., 1992, "Water Lubricated Pin on Disc Tests with Ceramics," *Wear*, Vol. 154, pp. 37~47.

Barth, M., Ensinger, W., Schröer, A. and Wolf, G. K. Low temperature generation of hard material coatings by reactive ion beam assisted deposition, in : T.S. Sudarshan, D.G. Bhat (EDS.), Surface Modification Technologies III, TMS, Warrendale, 1990, p. 195.

Cho, J. G. and Choi, M. S., 1994, "An Experimental Study of the Modified Chemical Vapor Deposition Process — Temperature Distribution and Particle Deposition Measurements," *Transactions of the Korean Society of Mechanical Engineers*, Vol. 18, No. 11, pp. 677~682.

Cuomo, J. J. and Rossnagel, S. M. Kaufman, H. R. (eds.), Handbook of Ion Beam Processing and Technology, Noyes, Park Ridge, NJ, 1989.

Dearnaley, G. and Hartley, N. E. W. Thin Solid Films 54 (1978) 215.

Ensinger, W. and Schrber, A., 1998, "Ion Beam Assisted Deposition of Carbide and Carbonitride of Titanium at low Substrate Temperature," Surface and Coatings Technology 103-104, pp. 168~172.

Garside, B. L., Mter. Sci. Eng. A 13(11) (1991) 179.

Halling, J., 1983, "Thin Solid Films," 108, pp. 103~115.

Halling, J., 1983, Principles of Tribology, The MacMillan Press. Ltd.

Hirvonen, J. P., Mater. Sci. Rep. 6 (1991) 215.

Hisakado, T., Suda, H. and Watanabe, H., 1992, "The Friction and Wear Mechanisms Between Ceramics and Metals," *Wear*, Vol. 155, pp. 251~268.

Hocking, M. G., Vasantasree, V. and Sidky, P. S., 1989, "Metallic & Ceramic Coating, Longman Scientific & Technical,"

Holmberg, K., Andersson, P. and Valli, J., 1987, "Three Body Interaction in Metal-ceramic and Ceramic-ceramic Contacts," *Proc. 14th* Leeds-Lyon Symp. on Tribology, pp. 227~235.

Honlmberg, K. and Matthews, A., 1994, "Coatings Technology Properties. Techniques and Applications in Surface Engineering," pp. 382~386.

Hubler, G. K., Mater. Sci. Eng. A115 (1994) 181.

Lee, K. H. and Kim, K. W., 1994, "Effect of Humidity and Sliding Speed on the Wear Behavior of Silicon Nitride Ceramics," *Transactions of the Korean Society of Mechanical Engineers*, Vol. 18, No. 10, pp. 2597~2605.

Lee, Y. M. Son, H. S. and Kim, J. Y., 1996, "Enhancenent of wear Resistance of TiN Coated High Speed Steel Tools Through Improving Some Coating Processes," *Journal of the Korean society of Precision Engineering*, Vol. 13, No. 11, pp. 32~37.

Oliver, W. C., Hutchings, R. and Pethica, J. B., Metall. Trans. A 159 (1984) 2221.

Peterson, M. B. and Winer, W. O., 1980, "Wear Control Handbook," *The Am. Soc. Mech. Eng.*, p. 894.

Rhee, B. G., 1995, "A Study on the Lubricational Characteristics with Compound Additives of Engine Oil," *Journal of the Korean Society of Tribologist and Lubrication Engineers*, Vol. 11, No. 1, pp. 50~57.

Ruck, D. M., Schmidt, H., Angert, N., Yoda, O., Aoki, Y. and Naramoto, H., in : Proceedings of the Ninth International Conference on Ion Beam Modification of Materials, Canberra, Australia, 5-10 February 1995, p. 1016.

Schröer, A. and Ensinger, W., Surf. Coat. Technol. 84 (1996) 448.

Sioshansi, P., Oliver, R. W., Matthews, F. D. and Vac. Sci. J., Technol. A 3(6) (1985) 2670.

Smidt, F. A., Int. Mater. Rev. 35 (1990) 62.

Song, G. and Ahn, H. S., 1990, "Wear Characteristics of TiN Coating by Plasma Enhanced CVD," *Journal of the KSLE*, Vol. 6, No. 1, pp. 116~125.

Tai, C. N., Koh, E. and Akari. K., 1990. "Macroparticles on TiN Films Prepard by the Arc Ion plating Process," *Surface and coatings Technology*, 43/44, pp. 324~335.

Vyskocil, J. and Musil, J., 1990, "Arc Evaporation of Hard Coating : Process and Film Properties," *Surface and Coatings Technology*, 43/44, pp. 299~311.