Journal of Metals, Materials and Minerals, Vol.21 No.1 pp.115-119, 2011

Effect of Nitrogen Partial Pressure on Characteristic and Mechanical Properties of Hard Coating TiAlN Film

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Abstract

(Ti,Al)N coating has been serving for the industry as cutting and forming tools. Compared with TiN film, TiAlN film significantly increases tool lifetime. Therefore, it reduces machine downtime and increases productivity. In this work, (Ti,Al)N film was deposited on cold-work tool steel (SKD 11) using PVD cathodic arc system. The sintered Ti-Al target with composition of (at%) 50 Ti and 50 Al was used as a cathode. The deposition bias voltage, bias arc current and deposition time were set at 100 V, 70 A and 90 minutes, respectively. Nitrogen gas was purged into the system with the applied pressure of 1, 1.5 and 2 Pa, respectively. After coating, the film was characterized for crystal structures and mechanical properties. It was found that all films have the same crystal structures of $Ti_{0.5}Al_{0.5}N$ and $TiN_{0.5}$ with the thickness in the range of 2-3 μm. Moreover, the film prepared at N_2 pressure of 1.5 Pa possesses the highest hardness (48 GPa), high adhesion strength (>150 N), and good adhesion (HF1 class). The difference of hardness and adhesion of the film was found to be resulted by size and dispersion of macroparticles.

Key words: (Ti,Al)N film, PVD, Cathodic Arc

Introduction

In the tooling and machining area, hard coatings are widely applied to prolong their service life and productivity. PVD cathodic arc plasma deposition is one of the promising techniques with high deposition rate to develop various advance materials for hard coating surface. The single-metal nitrides such as TiN, CrN, and ZrN are known as the first generation of PVD hard coating for machining tool. These nitrides possess higher hardness than of high speed steel and cemented carbide. However, the mechanical properties of these single metal nitrides are deteriorated at high temperature. To overcome these limitations, new types of coatings such as (Ti,Al)N [(1-3,9)], (Ti,Zr)N [(4)], (Ti,Si)N [(5,6)] etc. are being developed. In recent years, (Ti,Al)N films have drawn much attention because the ternary solid solution enhances the oxidation resistance at elevated temperatures as well as good wear resistance, hardness and lowers coefficient of friction [(7,8)]. The properties of (Ti,Al)N film can be controlled by various deposition parameters. The microstructure and mechanical properties of the (Ti,Al)N film are most dependent on bias voltage, nitrogen pressure and the aluminum to titanium ratio [(2,3,10,11,13,14)]. The phase composition of (Ti,Al)N film strongly depends on the overall aluminum contents which results in the different values of film hardness [(2)]. The effect of nitrogen partial pressure on the microstructure and surface roughness of the coating was also reported [(10-12)]. However, very few studies on the influence of nitrogen partial pressure on the film adhesion were reported.

In this study, Ti-Al sintered target with composition of (at%) 50 Ti and 50 Al was used as a cathode. The (Ti,Al)N film was deposited on the top of TiN interlayer coated over the SKD11 tool steel substrate using different nitrogen pressures. The results of characterization of these films of phase structure, surface morphology, hardness and adhesion behavior are presented.

Materials and Experimental Procedures

The sintered Ti-Al alloy having atomic ratios (Ti:Al) of 50:50 made by Thailand Institute

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of Scientific and Technological Research (TISTR) was used as target in this study. SKD11 (modified cold-work tool steel) with hardness approximately of 60 HRC was used as a substrate. The substrate size is 32-mm in diameter with thickness of 5 mm. Before coating, the substrates were mirror-polished, cleaned in an ultrasonic bath with alcohol and then dried in hot air. The targets and substrates were later fixed into PVD hard coating machine (NS-1, NanoShield PVD Hard Coating, Thailand). The PVD cathodic arc plasma deposition (CAPD) was processed in vacuum chamber. The substrates were further cleaned in vacuum using Ar glow discharge and titanium plasma. The metallic glue TiN interlayer were done with the same metal plasma sources for better adhesion. The deposition voltage and arc current were set at 100 V and 70 A, respectively. Nitrogen gas was supplied into the chamber at various values of pressure, which are 1, 1.5, 2 Pa and the coating time was fixed at 90 minutes. After deposition, Calotest (CSM Instruments, Switzerland) was used to determine the films thickness. Coating adhesion was initially evaluated by Rockwell C indentation (VDI-3198 German industrial standard) with a load of 150 kg. Subsequently, the indentation damage is examined under an optical microscope (Olympus, Japan and LOMO, Russia). A surface roughness analyzer (DIAVITE DH-7) and FE-SEM (JEOL JSM-6340F) were used to measure average surface roughness and investigated surface morphology of the coatings. X-ray diffractometry (Brukers D8) was used to identify the film phase structure using Cu Kα radiation at 40 kV and 30 mA with a low incident angle of 1° and the scanning angular (2θ) ranging from 30° to 90° with scanning rate of 2°/min. Hardness and adhesion of the films were characterized by Nanoindentation and Revetest Scratch Tester.

Results and Discussion

Ti-Al sintered target (50 at% Ti, 50 at% Al) has a bulk density of 3.56 g/cm^3 and the phase components, analyzed by XRD, consist of TiAl, $TiAl_2$, Ti, and Al. This target was used as a cathode in the PVD system to produce the hard coating film. The film thicknesses, as shown in Figure 1(a) were measured from the optical micrographs (Figure 1(b)) of the samples after experienced the Calotest. The film thicknesses, including the (Ti,Al)N top layer and TiN interlayer were approximately in the range of 1.96-3.16 µm. The TiN interlayer (gold color) was very thin around 0.4 µm. It was found that the film thickness decreased with an increase of N_2 pressure. This trend may be explained by difference of the energy of ions reacting the substrate. When the reactive gas pressure increases, a number of collisions of gas atoms increases. As a result the mean free paths and ions kinetic energy decreases. This phenomenon lowers deposit rate and subsequently lowers film thickness [11].

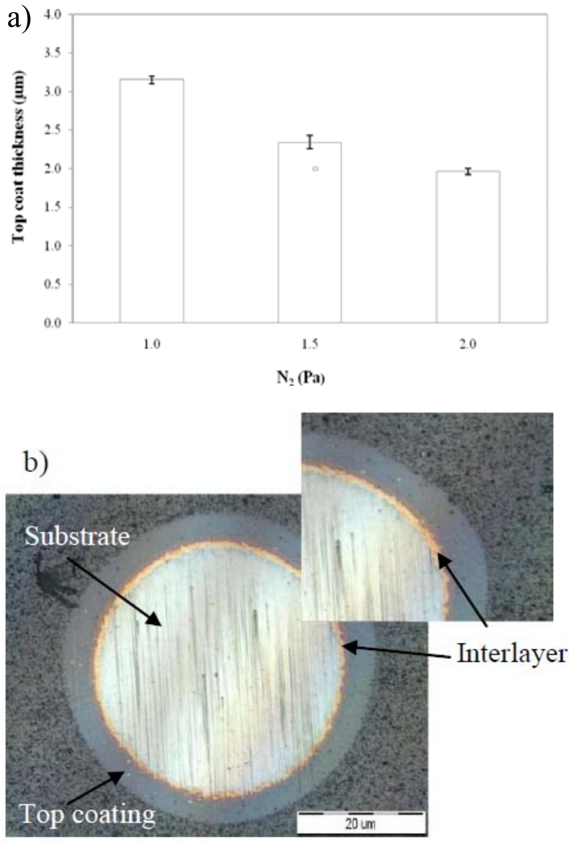

Figure 1. (a) The film thickness of samples coated by PVD using different N_2 pressure. (b) Optical micrograph of calotested sample.

$Ti_{0.5}Al_{0.5}N$ and $TiN_{0.5}$ phases characterized by XRD, were found for all films as shown in Figure 2. The peaks of $Ti_{0.5}Al_{0.5}N$ for specimen prepared at 1.5 Pa are higher than those of 1 and 2 Pa specimens, this is due to the film can be formed more $Ti_{0.5}Al_{0.5}N$ structure at N_2 partial pressure of 1.5 Pa. The surface roughness and SEM images of films deposited at various nitrogen pressures are shown in Figures 3 and 4, respectively. It was found that the coating surface was covered with a large number of macroparticles which cause the increase of surface roughness. The coatings deposited at 1.5 and 2 Pa show lower number of macroparticles than that deposited at 1 Pa. The number of these particles decrease with increasing the reactive gas pressure. As nitrogen pressure increases, the ion energy to the substrate decreases because the ions experience more collisions during their flight before reaching the substrate and then a decrease resulting in of kinetic energy. A similar dependence was also noticed by Bujak et al. [11].

Figure 5 shows the influence of nitrogen pressure on hardness and Young's modulus of (Ti, Al)N films. The film hardness of all samples is around 38-48 GPa. The samples prepared at 1.0 and 1.5 Pa N_2 exhibit superhard hardness (exceed 40 GPa). The film deposited at 1.5 Pa N_2 presents the highest hardness which results from the highly phase of $Ti_{0.5}Al_{0.5}N$ structure as mention earlier in Figure 2.

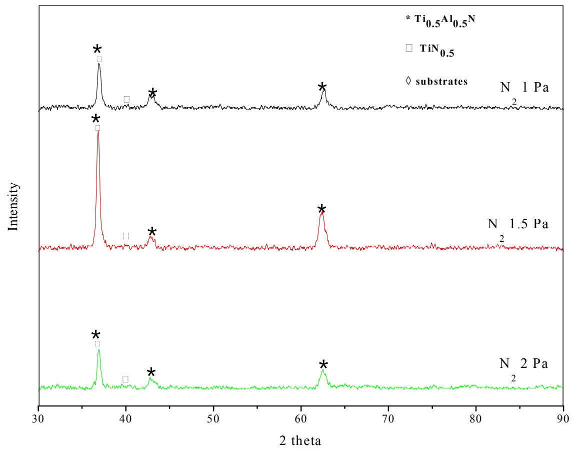

Figure 2. XRD patterns of the as-deposited coatings with nitrogen pressure of 1 Pa, 1.5 Pa and 2 Pa.

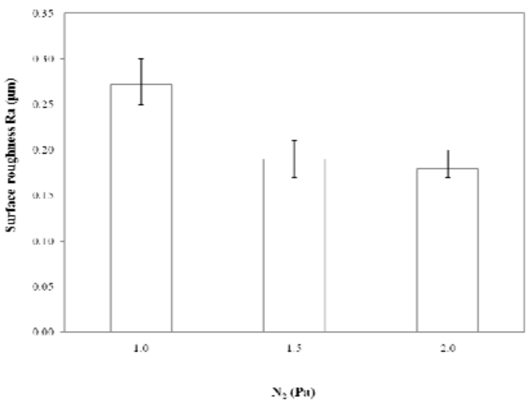

Figure 3. The surface roughness of samples coated by PVD using different targets and N_2 pressure.

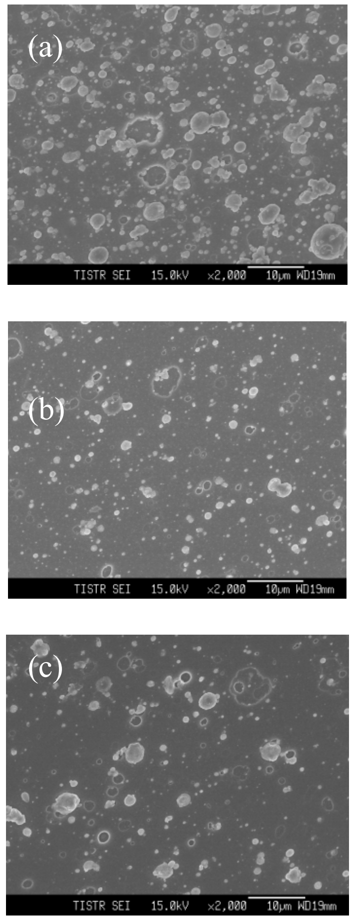

Figure 4. Surface morphology of the films deposited at difference N_2 pressure (a) 1 Pa (b) 1.5 Pa and (c) 2 Pa.

The adhesion of film to substrate tested from Rockwell C indentation is classified as HF1 for all films (deposit at 1, 1.5, 2 Pa N_2). In the HF1class, it can be explained that fracture is not found around the dent and the films have good adhesion (Figure 6). Scratch test is also used to investigate the adhesion of the film. It was found that the films deposited at 1 and 1.5 Pa have very good adhesion strength with the scratch load over 150 N (Table 1). These excellent properties in hardness, Young's modulus, and adhesion strength may be resulted from the reinforcement of small particles (1-2 μm in diameter), which uniformly disperse over the film matrix. On the other hand, system with coating condition at 2 Pa has high concentration of nitrogen during deposition. These nitrogen atoms may diffuse and forms nitrogen gas, show as voids in the film and in the interface between the films and the substrate [15]. As a result, the (Ti,Al)N film deposited at 2 Pa has poor adhesion to the substrate.

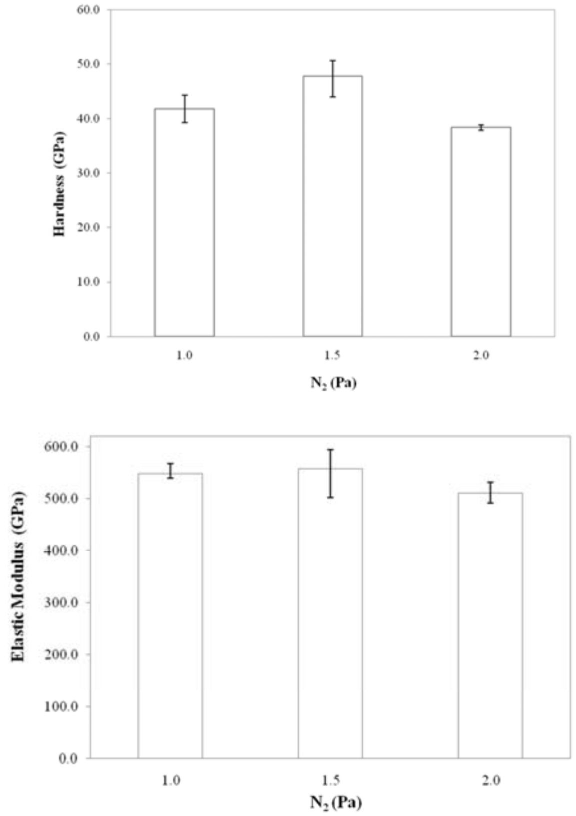

Figure 5. Hardness and Young's modulus, test by nanoindentation, of (Ti, Al)N films deposited at different N_2 pressure.

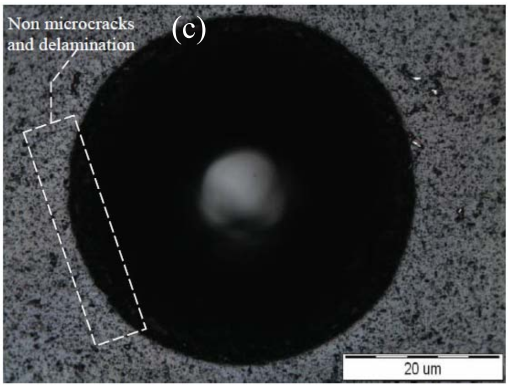

Figure 6. The image of Rockwell C indentation classified as HF1 for all films.

Table 1. Adhesion of the films.

N_2 (Pa)	Rockwell Adhesion Test VDI 3198	Loading at full delamination from Scratch test (N)
1.0	HF 1	>150
1.5	HF 1	>150
2.0	HF 1	128.43±3.95

Conclusions

In this study, $Ti_{0.5}Al_{0.5}N$ film with $TiN_{0.5}$ interlayer can be produced from PVD cathodic arc coating method using Ti-Al sintered target and N_2 pressure between 1-2 Pa. The films thicknesses were in the range of 1.96-3.16 μm. The film deposited at 1.5 Pa N_2, compared with film prepared at 1.0, 2.0 Pa, has the highest hardness due to the higher phase structure of $Ti_{0.5}Al_{0.5}N$. A good adhesion strength is caused by uniformly dispersion of small particles all over the films. Maximum value of the critical failure load (>150 N) was found for the coating deposited at the nitrogen pressure of 1 and 1.5 Pa. These coatings have very good adhesion strength. On the other hand, minimum critical failure load (Lc ≈ 128.43 N) were found for coatings deposited at 2 Pa.

Acknowledgements

The supports from Thailand Research Fund (TRF), Thailand Institute of Scientific and Technological Research (TISTR) and NanoShield PVD Hard Coating are gratefully acknowledged. Furthermore, the authors would like to thank Dr. Dmitri A. Karpov (FSUE D. V. Efremov Scientific Research Institute of Electrophysical Apparatus, Russia) for an advice on target design, Mr. Gregory Favaro (CSM Instruments Switzerland) for Nanoindentation Hardness and Scratch Adhesion test and Dr. Nakorn Srisukhumbowornchai for his help on XRD.

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