STRUCTURE AND PROPERTIES OF AlCrN AND TiAlN THIN FILMS COATED ON GAS NITRIDED SKD61 STEEL BY CATHODIC ARC PLASMA DEPOSITION

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ABSTRACT

In this study, AlCrN and TiAlN hard thin films were deposited on nitrided SKD61 steel by cathodic arc plasma evaporation method. Structure and properties of thin films were investigated by X-ray diffraction, optical microscope, stereo microscope, microhardness test, adhesion VDI 3198 standard, and corrossion resistance test. The results revealed that the a duplex structure of AlCrN (~ 2.2μm) or TiAlN (~ 2.5μm) and nitrided layers (~ 80μm) were formed on the SKD61 steel. The AlCrN and TiAlN thin films were composed of (AlN + CrN) and (AlN + $TiN_{0.3}$) phases, respectively. The adhesion of AlCrN and TiAlN films on the nitrided steel was very good. The surface hardness and corrossion resistance of stainless steel were improved by duplex treatment process of cathodic arc plasma deposition and nitriding.

Keywords: *AlCrN, TiAlN, cathodic arc plasma evaporation, corrosion resistance, corrosion resistance, hardness, hard thin films.*

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1. INTRODUCTION

Anticorrosion and wear resistance film produced by cathodic arc deposition has been widely applied in industry because of its outstanding advantages including density, uniformity, high deposition rate and adhesion. In addition, using this method, it is possible to deposit various types of films including single componet (TiN [1], CrN [2, 3], ZrN [4]), multicomponent (CrAlN [5], TiAlN [3, 5], TiAlSiN [6]), single layer, multilayer [7]... Among the protective films mentioned above, titanium ntride (TiN) and chromium nitride (CrN) are two the most common coatings fabricated by cathodic arc deposition method [8]. However, in some applications such as metal castings mold (aluminum, zinc) working at high temperatures up to 750°C [9], these two coatings cannot be used because of their low oxidation resistance. Several studies [10-13] have reported that the addition of Al in TiN and CrN can improve oxidation resistance up to 850°C due to structure refinement [10]. There for, TiAlN and CrAlN coating deposited by cathodic arc deposition have been studied and applied to materials like ceramic, Ti alloy and stainless steel [10, 13].

It should be noted that applying a hard coating (> 1500HV) to a substrate with a low hardness (500 - 600HV) will lead to plastic deformation of the coating under high intensity loading and reduce wear resistance of the coating/substrate system [14]. An advanced solution currently in use is duplex treatments consisting of nitriding and PVD technology. Nitriding technology creates iron nitride phases with high hardness and corrosion resistance on the steel surface, thereby forming a hardness gradient from the coating surface to steel substrate [15].

TiN, CrN and TiAlN films deposited on plasma nitride steel exhibit better anticorrosion, wear resistance, and adhesion than those coated on steel substrate without plasma nitriding [14-16].

Although duplex surface treatments combining nitriding and PVD coatings have been widely reported, most existing studies focus predominantly on plasma nitrided substrates. To date, there is a lack of systematic investigations on PVD coatings deposited on gas-nitrided steels. In Vietnam, gas nitriding remains the most widely applied surface treatment technology due to its simpler process control and lower investment cost compared with plasma nitriding. Despite its technological advantages, plasma nitriding requires sophisticated equipment and precise control, which limits its applicability in many mechanical manufacturing and heat-treatment companies in Vietnam. Therefore, the present study provides a novel and practically significant contribution by investigating AlCrN and TiAlN PVD duplex coatings on gas-nitrided SKD61 steel, offering an industrially feasible approach to enhance surface hardness, adhesion, and corrosion resistance.

2. EXPERIMENTAL PROCEDURE

Materials and sample preparations: SKD61 steel bar supplied by Daido steel (Japan) with chemical composition (in wt%) of of 0.387% C, 5.413% Cr, 1.035% Si, 0.758% V, 1.182% Mo and 0.441% Mn was cut into small samples with size of Ø22×7mm (diameter × thick). The steel samples were heat treated according to the process including quenching (1030°C/30 min) and double tempering 580°C/2 hours + 580°C/2 hours) in a vacuum furnace (Turbo2 Treater M). The average hardness after heat treatment is about 46 - 48HRC (520 ± $20HV_{0,05}$). After the tempering, all samples was ground with sandpaper having grit size from P120 to P1200 and gas nitrided at temperature 550°C using NH_3 with decomposition rate of 65 ± 5 %. As the surface was oxidized, the nitride sample was denoted as H-N was ground and polished with diamond power of 1µm grain size to remove the oxidation layer on the surface. Before deposition the nitride substrate (denoted H-N-0) was subjected to ultrasonic cleaning using alcohol for 30 min. AlCrN and TiAlN films was deposited by cathodic arc deposition method using Bal 1200 (model 1995) with parameters presented in Table 1.

Table 1. PVD coating parameters

Parameters	AlCrN	TiAlN
Target	AlCr	TiAl
Working pressure (torr)	5×10^{-3}	5×10^{-3}
Target current (A)	100	100
Deposition rate (µm/min)	0.02	0.02
Bios voltage (V)	-100	-100
Coating temperature (°C)	< 500	< 450

Investigation of surface properties*:* Keyence (VHX-7000) stereomicroscope was used to image the surface and imprint of HRC indenter to evaluate the adhesion. X-ray (Panalytical X'Pert Pro) is also used to determine the phase structure of the coating.

Coating thickness: The coating thickness was determined by using calotte grinding method as well as images observed in the optical microscope. This method lies in the fact that a hardened steel ball is rotated freely against the sample until the coating has been penetrated (penetration depth should be at least twice the coating thickness). By measuring wear scar in the shape of the circular projections coating thickness t can be calculated using the following Eq. (1):

$$t=\left[R^2-\left(\frac{d}{2}\right)^2\right]^{1/2}-\left[R^2-\left(\frac{D}{2}\right)^2\right]^{1/2} \quad (1)$$

where R is the ball steel radius, D is outer diameter, d inner diameter.

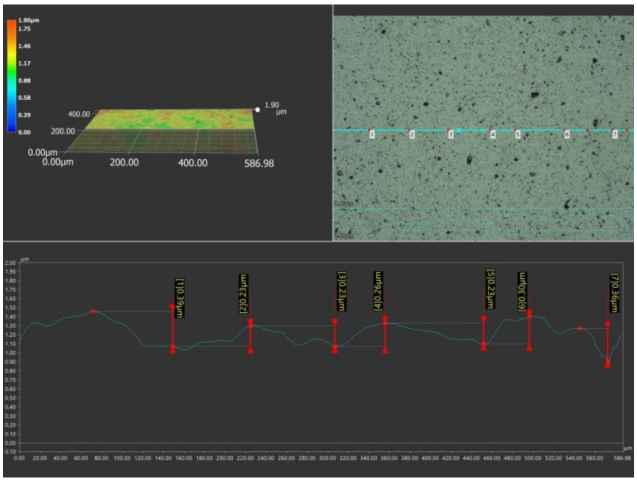
Coating hardness and adhesion: The surface hardness (HV0.1) of the coating was measured using Future (Tech Corp 700). According to the VDI 3198 standard the coating adhesion to the substrate is assessed through the images of imprints obtained by loading HRC indenter with 150kg onto surface sample using Keyence (VHX-7000) stereomicroscope, which can determine not only the shape but also the diameter and depth of the imprint and the roughness of sample surface.

Corrosion resistance: The corrosion resistance was evaluated in 3.5% NaCl solution using electrochemical workstation VSP 300 - France. Prior to electrochemical corrosion test, the samples were immersed in 3.5% NaCl solution for 30 min to stabilize open-circuit potential obtained with contact area of $1cm^2$. Potentiodynamic polarization tests were carried out from -800mV to -0.1mV with a scan rate of 1mV/s. After each potentiodynamic polarization test, the corrosion potential (E_{corr}) and the corrosion current density (I_{corr}) were determined from the Tafel plot.

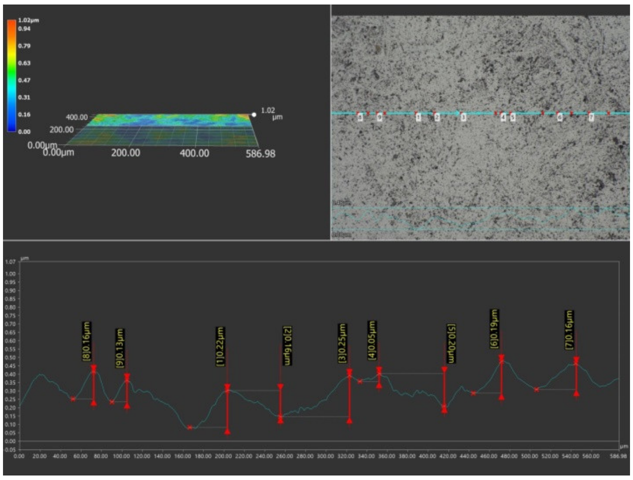
3. RESULTS AND DISCUSSION

3.1. Microstructure

The surface images of AlCrN and TiAlN coating are shown in Figure 1. The average surface roughness (Sa) of the AlCrN and TiAlN coatings, 0.07 and 0.06 respectively, are similar because two substrate surface was treated by the same process, while Rz are different due to uneven distribution of macroparticles (peaks) and valleys on the surface. The appearance of macroparticles related to the essence of the cathodic arc deposition method adversely affects the coating properties.



a) AlCrN

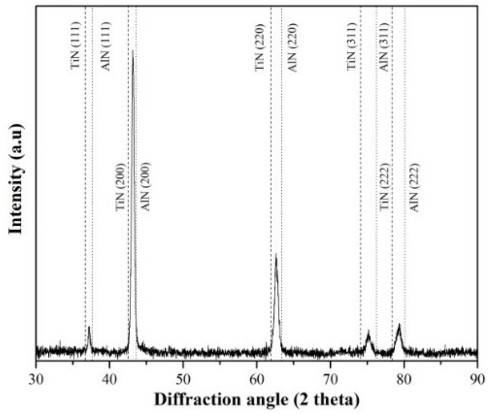


b) TiAlN

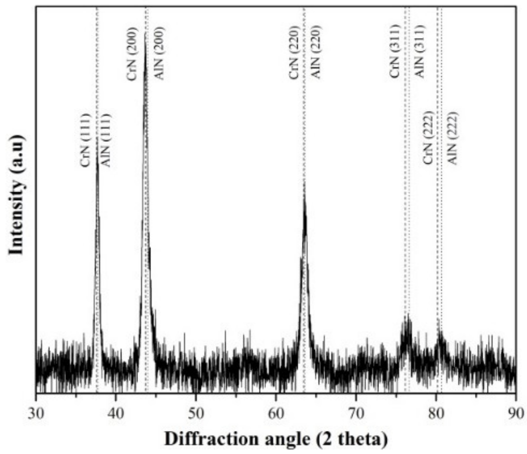
Figure 1. Surface image by optical microscope: a) AlCrN và b) TiAlN

Figure 2 shows that coated sample produced diffraction peaks, which matched with (111), (200), (220), (311), (222) planes of face-centered cubics (Al, Cr)N or (Ti, Al)N. In both cases these peaks lie between those of c-AlN (ICDD Card No. 00-025-1495) and c-CrN (ICDD Card No. 00-011-0065) or c-TiN (ICDD Card No. 00-038-1420) because TiAlN and AlCrN possess the same crystal structure as TiN and CrN due to replacing Cr and Ti atoms by Al atoms in nitride films resulting in the formation of Al-N bond and solid solutions Ti-Al-N or Cr- Al-N [17, 19]. However, if this substitution occurs strongly, it leads to a transition from the face centered cubic of nitrided structure to a hexagonal wurtzite structure of AlN (h-AlN) with low hardness [20]. In these experiments no appearance of wurtzite structure was observed, which may be due to the use of targets having Al:Cr and Ti:Al ratios of 50:50. As can be seen in Figure 2a, substitution of Ti with the covalent radius of 0.146nm by Al with the smaller atomic radius of 0.143nm caused shrinkage in the lattice parameter as well as generation of internal stress in the coated film. Thus, diffraction peaks shifted to higher angle, which indicated the presence of tensile stress [21]. This also happened for AlCrN film because diffraction peaks corresponding to (111), (200), (220), (311), (222) crystal planes of c-CrN and c-AlN phases are located close to each other so the displacement is not significant.

Experimental validation is conducted using a prototype platform equipped with the gyroscopic module, control electronics, and inertial sensors. During testing, the vehicle is subjected to manual disturbances while operating on a flat surface. The system demonstrates improved balance recovery and reduced oscillatory motion when the gyroscopic module is active. The experimental observations are consistent with simulation results, confirming the effectiveness of the proposed stabilization approach.



a)



b)

Figure 2. X-ray diffraction patterns: a) H-N; b) AlCrN; c) TiAlN

Practical implementation issues such as sensor noise, actuator delay, and vibration are also considered. Signal filtering is applied to sensor measurements to prevent excessive controller reaction. Proper tuning of control parameters is necessary to avoid overcompensation and ensure stable operation.

3.2. Coating thickness

The thickness of AlCrN and TiAlN films are 2.2µm and 2.5µm respectively by CGM test (calotte grinding method). Appying the formula (1) mentioned above:

$$t = \sqrt{20000^2 - 145^2/4} - \sqrt{20000^2 - 642^2/4} = 2.444784\mu m$$

Where: R = 2000µm, D = 624µm và d = 145µm, with D and d are determined as Figure 3.

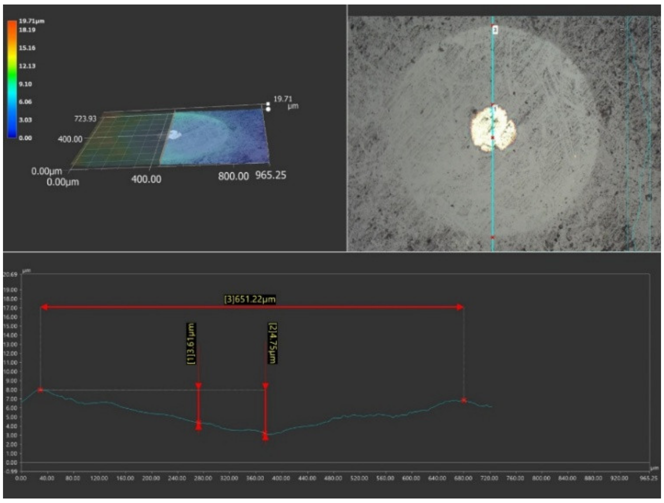


Figure 3. The image depicts the morphology of abrasion marks on a coated sample using the calotest method

3.3. Coating hardness and adhesion

The hardness of the nitride sample after coating with TiAlN and AlCrN films are 2750 and 2430HV$_{0.1}$, respectively, while nitride sample have only the hardness in the range of 950HV$_{0.1}$. Surface hardness of TiAlN and AlCrN films are lower than that reported in other studies, 2800 - 3300HV and 2500 - 3000HV, repectively due to the influence of the soft substrate on the measured hardness value [18, 22].

In this experiment according to VDI standard 3198, the AlCrN film has a higher adhesion, HF1, compared to the TiAlN film, HF2 (Figure 4). There are many factors, which affect the film adhesion. In which, according to the study [23], AlCrN film with preferential orientation in (111) and (200) planes show better density and adhesion to the substrate. In addition, the difference in adhesion may be due to the stress deviation between the substrate and the coating. AlCrN film has a hardness close to that of the substrate so the stress difference is lower and the adhesion is better.

a)

b)

Figure 4. Indenter imprints of adhesion test a) AlCrN và b) TiAlN

3.4. Corrosion resistance

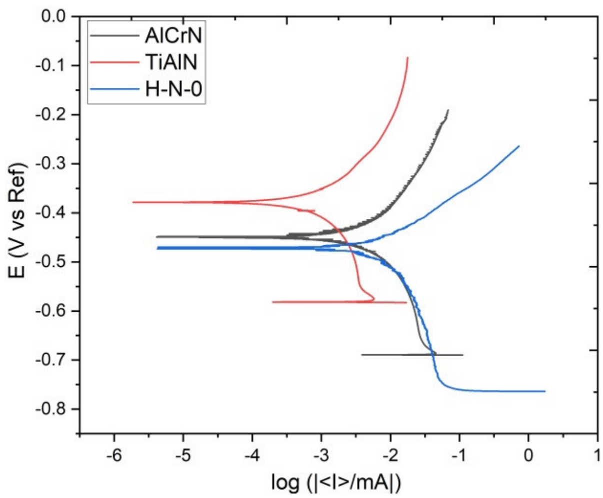


Figure 5. Electrochemical polarization curves

As shown in Figure 5 the coatings shifted the corrosion potential difference to more positive values, that is, the corrosion resistance is improved.

Table 2 shows the E_{corr} and I_{corr}, as well as the anodic (βa) and cathodic (βc) Tafel slopes for the nitride substrate and AlTiN, AlCrN coating sample.

Table 2. Corrosion parameters extracted by Tafel fitting on potentiodynamic polarization curves of the coatings

	Substrate	AlCrN	TiAlN
E_{corr}, V	-0,468	-0,449	-0,378
i_{corr}, μA/cm^2	7,823	4,237	0,908
β_a (mV/decade)	100.4	168.4	140.1
β_c (mV/decade)	238.8	160.3	196.1
P_i (%)	-	45.84	88.39

Specifically, the corrosion potential of H-N-0 increased from -0.468V_{SEC} to -0.449V_{SEC} with AlCrN coating and -0.378V_{SEC} with TiAlN coating. At the same time, the corrosion current density decreased from 7,823μA/cm^2 to 4,237μA/cm^2 and 0.908μA/cm^2 with AlCrN and TiAlN, respectively.

The efficiency P(%) of the coating as shown in Table 2 was calculated by Eq. (2) [24]:

$$P(\%) = \left[1 - \left(\frac{i_{corr}}{i^0_{corr}}\right)\right] \times 100 \quad (2)$$

Whre i_{corr} and i^0_{corr} indicate the corrosion current density of the film and substrate, respectively. It can be seem form Table 3 that AlTiN coating has efficiency of 88.9%. In comparison, the protective efficiency of AlCrN coating is only 45.84%.

Therefore, TiAlN coating has better corrosion resistance than AlCrN coating. This may be because the surface of the AlCrN-coated sample has pinholes, as shown in Figure 2, appeared due to the sloughing of macroparticles causing insufficient protection of the coating for nitride substrate.

4. CONCLUSION

TiAlN and AlCrN coatings on nitrided steel have good adhesion (HF1 and HF2) and surface hardness (2750 and 2430HV0.1, respectively) ensuring the required adhesion to parts operating in in conditions of high temperature and abrasive wear.

TiAlN and CrAlN coatings obtained by cathodic arc deposition with parameters such as bios voltage -100V, target: TiAl, AlCr, working pressure: 5×10^{-3} torr, have face centered cubic structure and preferred orientation in (111) for TiAlN and (111) and (200) for CrAlN.

Both TiAlN and AlCrN coatings improved corrosion resistance of nitrided steel, where AlCrN has better corrosion resistance.

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