

Characterization of TiAlBN Nanocomposite Coating deposited via Radio Frequency Magnetron Sputtering using Single Hot-Pressed Target

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Abstract. TiAlBN coatings have been deposited at varying bias voltage of 0, -60, and -150 V by radio frequency (RF) magnetron sputtering technique. A single hot-pressed Ti-Al-BN target was used for the deposition process. With glancing angle X-ray diffraction analysis (GAXRD), the nanocrystalline (nc-) (Ti,Al)N phase was identified. In addition, the existence of BN and TiB₂ amorphous (a-) phase were detected by X-ray photoelectron spectroscopy (XPS) analysis. Thus, the deposited TiAlBN coatings were confirmed as nc-(Ti,Al)N/a-BN/a-TiB₂ nanocomposite. On the other hand, it was found that optimum bias voltage used in present study is -60 V where the deposited TiAlBN coating exhibits an excellent adhesion quality. The adhesion quality of the coatings deposited at -60V bias voltage is classified as HF 1 evaluated using the Rockwell-C adhesion test method (developed by the Union of German Engineers).

Introduction

Recently, nanocomposite coatings had been studied extensively due to its superior mechanical properties. A nanocomposite coating can be defined as a coating consisting of very fine crystallites (<10 nm) embedded into amorphous matrix [1]. If a nanocomposite coating is prepared properly, its hardness may even surpasses the hardness of a diamond [2]. TiAlBN coating is one of the example of nanocomposite coating which exhibit a nc-(Ti,Al)N/a-BN structure.

The TiAlBN coating had seen to catch many attentions due to its excellent hardness, wear resistance, thermal stability, long-term stability, and drilling performance [3-9]. The coating can be deposited by various PVD techniques such as electron-beam (EB) evaporation [3,6], and direct current (DC) magnetron sputtering [5,8]. However, the deposition of the TiAlBN coating by radio frequency (RF) magnetron sputtering technique has received only limited attention [9].

By employing the RF magnetron sputtering technique, several benefits can be acquired compared to the EB evaporation technique. For example, a lower power requirement, and lower radiant heat load during processing [10]. In addition, coatings produced by RF magnetron sputtering were found to be denser and harder compared to DC magnetron sputtering [11]. Therefore, in this study, an attempt to deposit a nanocomposite TiAlBN coating was carried out by employing the RF magnetron sputtering technique. In addition, the most suitable bias voltage to produce the optimum coating characteristic (for present work) was determined based on the adhesion quality of the deposited TiAlBN coating. The nanocomposite was characterized using GAXRD and XPS analysis, while the adhesion quality is evaluated by Rockwell-C adhesion test.

Experimental Setup

The TiAlBN coatings were deposited on AISI 316 stainless steel and silicate glass substrate using PSP5004 SNTTEK radio frequency (RF) magnetron sputtering. The target material for the deposition process was made from a hot-pressed material consist of 70 wt.% Ti, 15 wt.% Al, and 15 wt.% BN powder mixture. The applied target power, deposition time, base pressure, and working pressure was set at 400W, 90 mins, $<5 \times 10^{-6}$ torr and 10 mtorr respectively. In addition, the nitrogen and argon gas flow was controlled at 5 and 15 sccm respectively. A set of coating was obtained with varying bias voltages at 0, -60, and -150 V.

The crystallographic phase, structure, and grain size of the coating were determined using PANalytical glancing angle X-ray diffraction (GAXRD) at fixed X-ray incidence angle of 2° . The grain size of the coating was calculated according to Scherrer formula. The amorphous phases in the TiAlBN coating were determined from X-ray photoelectron spectroscopy (XPS) spectra acquired using XSAM HS Kratos spectrometer based upon coating deposited at -60V bias voltage. Then, curve fitting was carried out implying Shirley background subtraction at B 1s, and N 1s peak area to identify the phases. In this analysis, only coating deposited at -60 V bias voltage was utilized due to the fact that variation in bias voltage had minor effect on coating's microstructure and chemical composition [8].

The coating adhesion is qualitatively evaluated using Rockwell-C adhesion test method developed by the Union of German Engineers [12]. For that purpose, hardness tester was employed. The indentations were made by diamond indenter at 150 kgf (using Mitutoyo HR-523 hardness tester) which will cause layer damage adjacent to the boundary of the indentation. Then the adhesion quality is classified according to HF 1 to HF 6 category, in which HF 1 to HF 4 is defined as sufficient adhesion whereas HF 5 and HF 6 represent insufficient adhesion [12].

Results and Discussion

Nanocomposite Structural Characterization. The GAXRD patterns of the deposited TiAlBN coatings at various bias voltages are shown in Fig. 1. The deposited coatings showed diffraction peaks attributed to TiN phase at peak (111), (200), and (220). The (300) and (222) TiN peaks at higher angles were very low in intensity. Apart from TiN phase, other possible peaks corresponding to phases such as Ti, Al, AlN, Al_3Ti , TiB_2 , BN [4,9] were not detected in the GAXRD patterns. This is due to the fact that these phases may exist as amorphous or not present at all. In addition, the calculated grain size determined via Scherrer equation was in a nano range in between 4.6 and 8.8 nm.

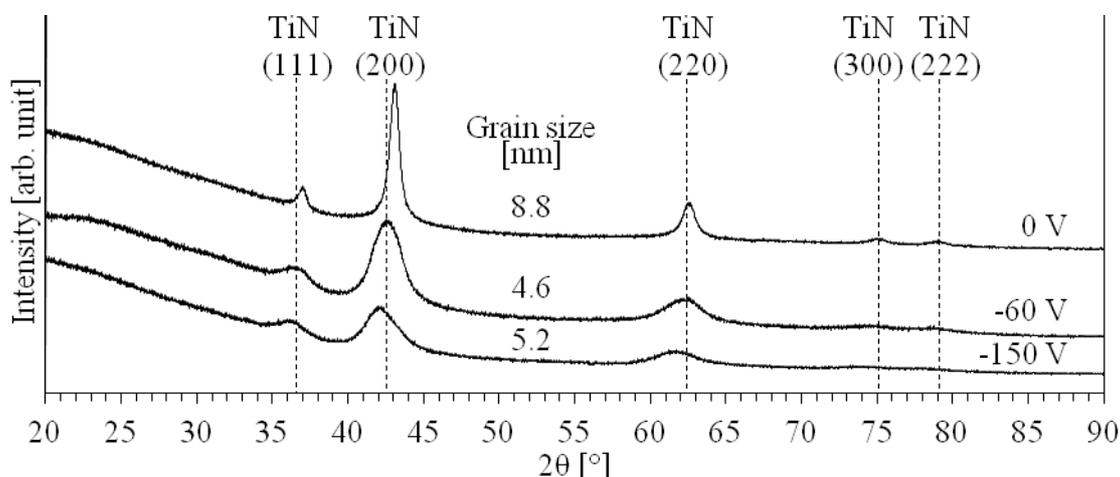


Fig. 1 GAXRD pattern and grain size of TiAlBN coating deposited at varying bias voltage

Based on the GAXRD pattern of the coating deposited at 0 V bias voltage, it should be noted that the diffracted peaks of the TiN phase shifted to the higher angle as compared to the reference peak position of pure TiN phase (represented by dotted lines). For example, at plane (200), the diffracted peak of the coating was positioned at 43.1° , whereas the reference peak was positioned at 42.6° . The peak shifting of the TiN phase to higher angles observed in the GAXRD pattern is attributed to the decrease of lattice parameter of the coating. The decrease of lattice parameter is related to the incorporation of aluminium atoms (in which its radius is smaller than titanium atoms) into the TiN lattice [8,14]. Therefore, it could be deduced that the TiN phase identified in the deposited coatings is representing the nanocrystalline (-nc) (Ti,Al)N phase.

It also can be observed that as the bias voltage increases, the diffraction peaks of the TiAlBN coatings shifted to lower angles. For example, the peaks at plane (200) shifted from 43.1 , 42.5 , to 42.2° for TiAlBN coating deposited at 0, -60, and -150 V respectively. This is due to the increase in intrinsic stress of the TiAlBN coating which results from enhanced ion bombardment when higher bias voltage was used [9]. In addition, broadening of the diffracted peaks can also be noted which is related to the reduction of crystallite size by increasing bias voltage.

Fig. 2 shows the curved fitted XPS spectra of the TiAlBN coating at B 1s and N 1s peak areas. At B 1s (Fig. 2a), the peak can be curve fitted into two main components attributed to BN and TiB_2 phase at 190.65 and 187.53 eV respectively [7]. Due to high reactivity of boron with oxygen, a small third component corresponding to B_2O_3 was also added in the B 1s peak. On the other hand, the N 1s peak (Fig. 2b) can be curve fitted into two components attributed to BN and (Ti,Al)N phase at 398.4 and 397.11 eV respectively [7]. The existence of the (Ti,Al)N phase detected through the XPS analysis is in agreement with the shifted diffraction peak during GAXRD analysis. It also can be concluded that the BN and TiB_2 exist as amorphous (a-) phase as it is not revealing in any diffraction peak in the GAXRD pattern. Thus, it can be suggested that the TiAlBN coating deposited at present work compose of nanocomposite structure in which nc-(Ti,Al)N is embedded into the a-BN and a- TiB_2 amorphous matrix (nc-(Ti,Al)N/a-BN/a- TiB_2).

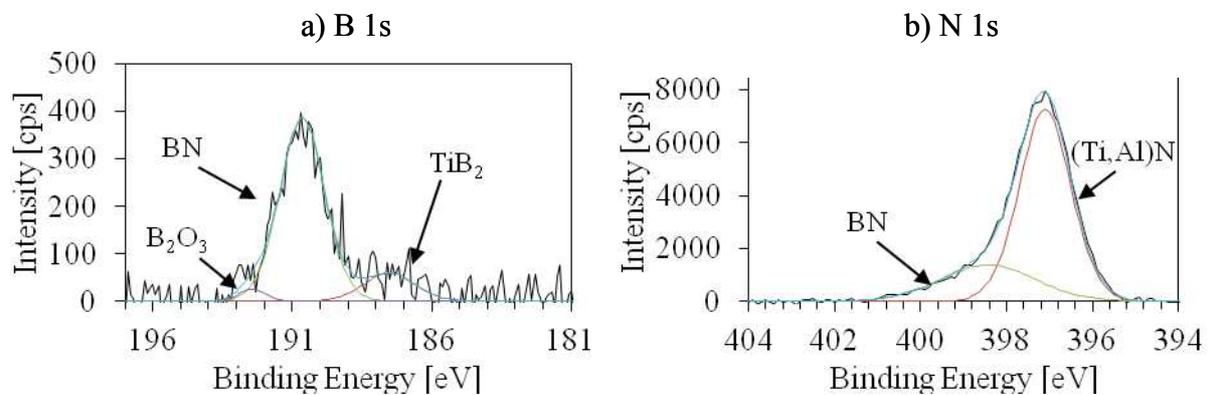
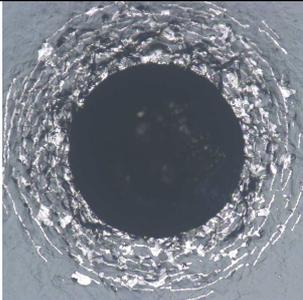
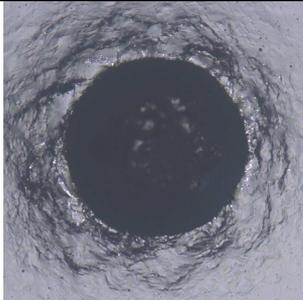
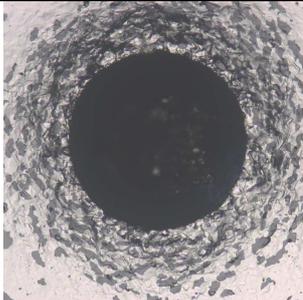


Fig. 2 XPS curve fitted spectra of TiAlBN coating at a) B 1s, and b) N 1s region

Adhesion Quality. The Rockwell-C adhesion test result on TiAlBN coating deposited at various bias voltage are shown in Table 1. It was observed that the coating deposited at -60 V showed better adhesion quality compared to the other samples. At 0 V bias voltage, intense radial cracks and delamination around the indentation can be identified. The adhesion quality of the coating can be classified as HF 5. However, as the bias voltage was increased to -60 V, significant improvement can be observed. At -60 V bias voltage, no radial crack and delamination was present. As such, the adhesion coating can be classified as HF 1. For coating deposited at -150 V, severe delamination around the indentation occurred. Thus the adhesion quality of the coating can be classified as HF 6.

Table 1 Rockwell-C adhesion test results of TiAlBN coating deposited at various bias voltage

Bias Voltage (V)	0	-60	-150
Micrograph Result			
Adhesion Quality	HF 5	HF 1	HF 6

Conclusion

The (Ti,Al)N/a-BN/a-TiB₂ nanocomposite coating was successfully deposited via RF magnetron sputtering technique at varying bias voltage using single hot-pressed target. The nanocomposite structure was evaluated by GAXRD and XPS analysis in which the presence of nc-(Ti,Al)N phase was determined from GAXRD pattern, and the a-BN and a-TiB₂ phases were confirmed from curve fitting of XPS spectra. It was also found that varying bias voltage significantly affects the adhesion quality of the deposited TiAlBN coating. At 0, and -150 V bias voltage, poor adhesion quality was shown (HF 5 and HF 6 respectively), whereas at -60 V bias voltage, the deposited coating exhibit an excellent adhesion quality (HF 1). Therefore, it can be concluded that -60 V bias voltage is the most suitable bias voltage used to deposit the TiAlBN nanocomposite coating in present work.

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