

# DEPOSITION OF TITANIUM ALUMINIUM NITRIDE THIN LAYER ON HIGH SPEED STEEL SUBSTRATE BY RADIO FREQUENCY SPUTTERING

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## ABSTRACT

High Speed Steel (HSS) has been widely used in manufacturing industry as one of cutting tool materials because of its good mechanical properties yet with a low price. However, since this material has a low thermal resistance property, it will limit its application when used in dry cutting process. Several methods have been used to improve the cutting performance of HSS in dry cutting. One of them was by growing a thin layer of hard coating on the contact surface of the cutting tool material. In this research, Titanium Aluminium Nitride layer were deposited on AISI M41 High Speed Steel substrate by using Radio Frequency (RF) Sputtering method. The aims of this study were to analyze the effect of variations of Aluminium surface area ratios (10, 20, 30, and 40 %) on the Titanium target and also to analyze the effect of deposition time (15, 30, and 45 minutes) on the composition, phase characterization and morphology of the thin layer that formed. The formation of TiAlN and AlN crystalline compounds were observed by X-Ray Diffraction method. A dense layer with a thickness range from 1.4 to 5.2  $\mu\text{m}$  was observed by using a Scanning Electron Microscopy. It was known that the deposition time affect the thickness and also the roughness of the layer. The topography images by Atomic Force Microscopy showed that the deposition time of 45 minutes produce the finest layer with the surface roughness of 10.8 nm.

**Keywords:** High Speed Steel, Titanium Aluminium Nitride, Radio Frequency Sputtering

## I. INTRODUCTION

Requirements for greater productivity and better quality of products in the manufacturing process increase the need for high performance cutting tools. Some mechanical properties that must be owned by these high performance cutting tools such as high hardness, high wear resistance, thermal stability and also high oxidation resistance. High Speed Steel (HSS) is one of the cutting tools that have been widely used in manufacturing industries. However, the performance of this material is not high enough to support an advance cutting processes such as dry cutting.

Physical Vapor Deposition (PVD) coatings, such as Titanium Aluminium Nitride (TiAlN) are widely used for numerous metal cutting applications due to their high hardness, low friction, wear, and oxidation resistance. [2] The addition of aluminium (Al) into this TiAlN layer has been known can increase its wear and oxidation properties through the formation of  $\text{Al}_2\text{O}_3$  dense layer when heated in elevated temperature [7]. This layer will act as a protective layer to prevent diffusion of oxygen into the covered surface [3] and also can lubricate the friction pair contact and isolate the coating underneath [6]. There have been many methods to synthesize TiAlN thin layer such as magnetron sputtering, arc evaporation, ion beam-assisted deposition and arc bonding sputtering [12].

In this research TiAlN thin layers were deposited on an AISI M41 HSS substrate by Radio Frequency magnetron sputtering. The effects of various surface area ratios of Al to Ti and deposition time on the composition, layer thickness, phase, and roughness of TiAlN layer were investigated. The phase and morphology characterization of the TiAlN layer were examined by X-Ray Diffraction

(XRD) and Scanning Electron Microscopy method, respectively. The composition of the layers were determined by using Energy Dispersive X-Ray spectroscopy (EDX). Atomic Force Microscopy (AFM) was used to identify surface topology and roughness of the TiAlN layer.

## II. MATERIALS AND METHODS

### Materials

The substrate material employed for this research was AISI M41 High Speed Steel with the composition of alloys given in Table 1 [1]. Before deposition, the substrates were abraded with SiC paper and then cleaned in alcohol for 10 min. The target used for TiAlN deposition were pure (99.99 %) titanium 75 mm in diameter and pure aluminium (99.00 %). The aluminium targets then covered titanium targets to achieve various surface area ratios of Al to Ti which were used in this research (10, 20, 30, and 40 %).

**Table-1.** Composition of AISI M41

Element	C	Si	Cr	V	W	Mo	Co
Amount (%)	1.10	0.33	4.13	2.00	6.63	3.75	8.25

### Thin Layer Deposition

Before sputtering, the chamber was evacuated down to base pressure of  $9 \times 10^{-6}$  Torr by using both mechanical and diffusion pumps. Once the vacuum was

achieved, argon (Ar) was introduced into the chamber in

A radiofrequency power of 210 W was used with 1100 V of voltage. After the plasma was formed, nitrogen was introduced into the chamber and nitriding began. The total working pressure was  $1.8 \times 10^{-2}$  Torr and the deposition time were set for 15, 30, and 45 minutes.

### Thin Layer characterization

The compositions of the layers as well as the morphological measurements and layer thickness were identified with SEM FEI Inspect S50 scanning electron microscope which was also equipped with an Energy Dispersive X-Ray Spectroscopy to obtain the chemical composition of the TiAlN layer. The X-Ray Diffraction (XRD) was used to study the phase and crystal structure of TiAlN layer, using Phillips Analytical equipment and a Cu-K $\alpha_1$  with a wavelength of 1.54056 Å. The topography characterization and roughness were measured by NEOS N8 Atomic Force Microscopy equipment.

### III. RESULT AND DISCUSSION

The composition of the TiAlN thin layer were identified by EDX. Table-2 summarizes the compositions and thickness of TiAlN thin layer deposited with various surface area ratios of Al on the Ti target and also various deposition time. It indicates with increasing aluminium area of the sputtering target, the aluminium content in the TiAlN layer gradually increases due to the sputtering yield of aluminium which is higher than titanium [10]. As the surface of the Ti target is replaced by Al disks, there are more sputtered atoms arriving at the substrate [5]. While the deposition time does not significantly impact the TiAlN composition.

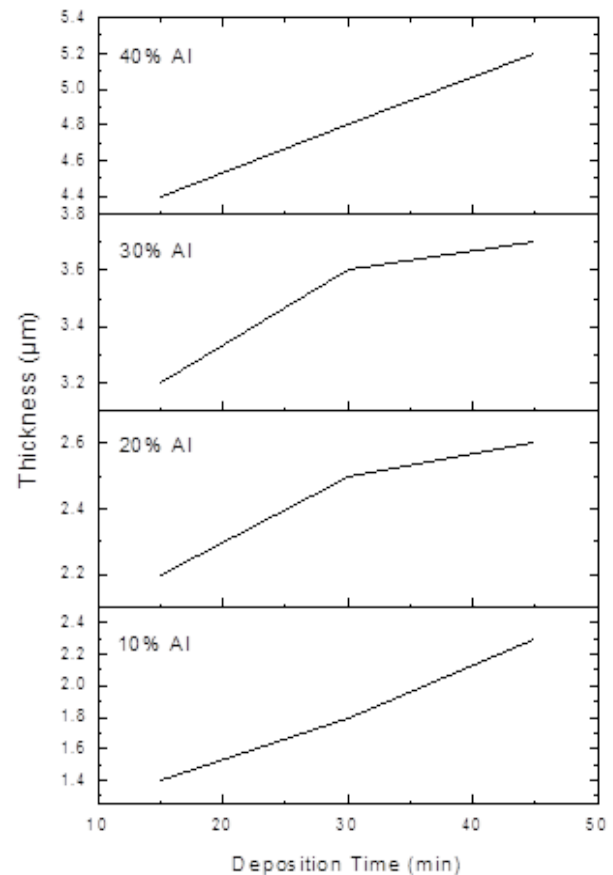
**Table-2.** Composition of TiAlN thin layer

Surface Ratio of Al (%)	Deposition Time (min)	Composition	Thickness ( $\mu\text{m}$ )
10	15	Ti <sub>47</sub> Al <sub>53</sub> N	1.4
	30	Ti <sub>53</sub> Al <sub>47</sub> N	1.8
	45	Ti <sub>57</sub> Al <sub>43</sub> N	2.3
20	15	Ti <sub>25</sub> Al <sub>75</sub> N	2.2
	30	Ti <sub>29</sub> Al <sub>71</sub> N	2.5
	45	Ti <sub>30</sub> Al <sub>70</sub> N	2.6
30	15	Ti <sub>10</sub> Al <sub>90</sub> N	3.2
	30	Ti <sub>11</sub> Al <sub>89</sub> N	3.6
	45	Ti <sub>11</sub> Al <sub>89</sub> N	3.7
40	15	Ti <sub>5</sub> Al <sub>95</sub> N	4.4
	30	Ti <sub>7</sub> Al <sub>93</sub> N	4.8
	45	Ti <sub>7</sub> Al <sub>93</sub> N	5.2

Deposition of the ternary compounds by physical vapor deposition usually requires an alloy target like smelting targets or hot-pressed powder targets [5]. But several reports show that these methods have many difficulties and problems such as potential of oxidation and high melting temperature requirement. However in this research it is observed a mosaic method by put aluminium plate on the titanium target surface provides an

order to achieve a pressure of  $0.9 \times 10^{-2}$  Torr. easy and convenient approach to the fabrication of compound targets.

The cross-section SEM image was used to determine the thickness of the TiAlN layer. Figure-1 shows that the thickness of the TiAlN layer increases from 1.4  $\mu\text{m}$  for 10 % Al with 15 minutes of deposition time to 5.2  $\mu\text{m}$  thick for 40 % Al with 45 minutes of deposition time. The increase in thickness is attributed to the composition of TiAlN and also the deposition time. As the deposition time become longer, it increases the growth time of TiAlN layer on the substrate.

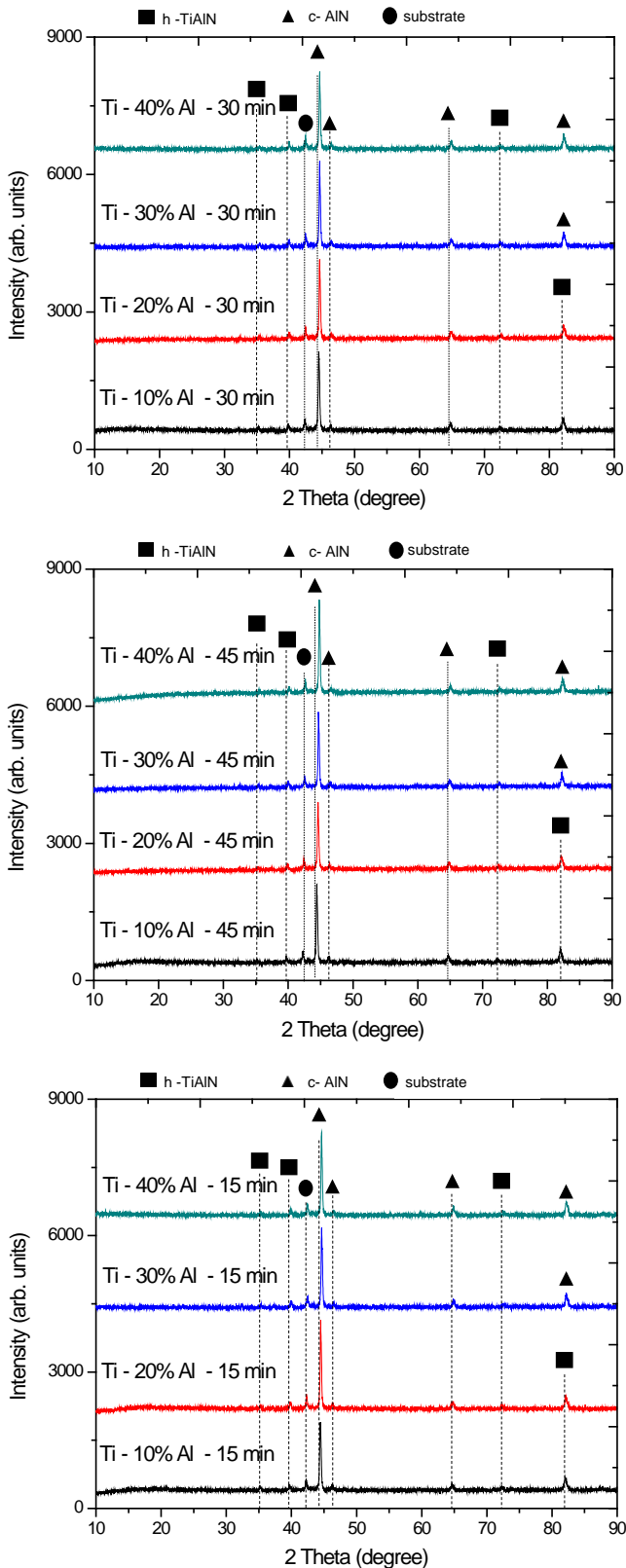


**Figure-1.** The thickness of TiAlN with various surface area ratios of Al and deposition time

The X-ray diffraction patterns of the TiAlN layer with various surface ratio of Al and deposition time of 15, 30, and 45 minutes are shown in Figure-2. Two primary crystalline phases, cubic structured AlN (c-AlN) and hexagonal structured TiAlN (h-TiAlN) can be identified in all composition. The 10 % Al sample shows the characteristic peak of the (200) AlN phase which slightly shift toward higher  $2\theta$  value due to the addition of Al surface ratio on the Ti target. This may indicate a change in the arrangement of atoms in the AlN crystal. According to Bragg’s Law, the higher value of diffraction peak will make the interplanar spacing (d) become much smaller. This can be caused by the stress release in the crystal that is characterized by impairment of micro strain in the crystal.

Gonzalez et al. [4] reported that with 20 % Al, the diffraction pattern show a composite material composed of AlN and TiN phases, with a respective grain

size of 14.3 nm and 2.9 nm. Tanaka et al. [8] reported that with an increase in Al content the film of  $Ti_{31}Al_{69}N$  exhibited mixed phases of hexagonal and cubic structures. While Zhou et al. [11] observed that  $(Ti_{1-x}Al_xN)$  coatings show a B1 NaCl type cubic structure for  $0 < x < 0.6$  and a mixed structure of B1 NaCl type cubic plus B4 wurzite type hexagonal AlN structure for  $0.6 < x < 0.8$  and only hexagonal structure for  $x > 0.8$ .



**Figure-2.** X-Ray diffraction patterns of TiAlN with various surface area ratios of Al and deposition time

It is observed that all the diffraction peaks of TiAlN are shifted to higher  $2\theta$  angles when the Al percentage is increased, as indicated in Figure-1. This implies that the lattice spacing of former TiN cell is decreased with increasing aluminium content due to the substitution of titanium atoms with aluminium atoms in TiN cell. In general the interstitial site should be large enough to accommodate the interstitial atom, otherwise the presence of the interstitial atom will expand the metal host lattice to the point where metal-metal interactions become weak and the structure will lose its stability.[9]

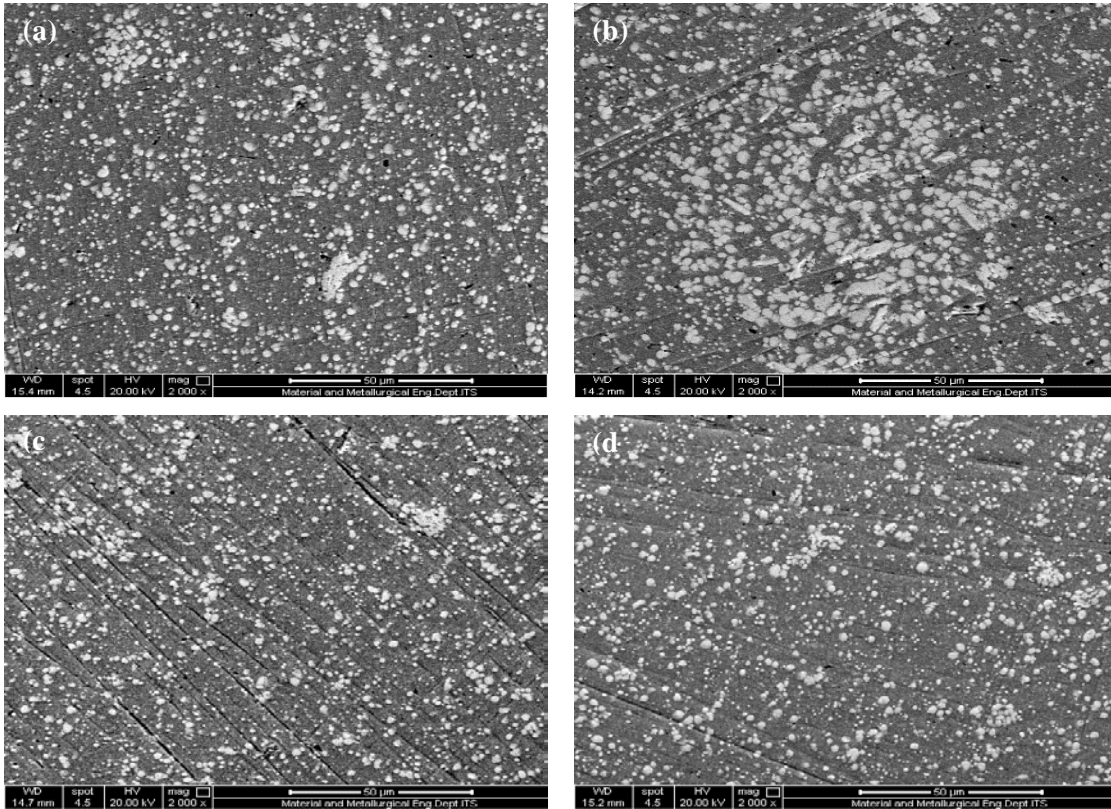
In Figure-3, Figure-4, and Figure-5 we can see SEM images of TiAlN thin layer surface morphology, magnified 2000 times. It is observed that the substrate is not fully covered by TiAlN layer with only ~70 % area are covered. Since the distance of substrate material and sputtering target was too close when the sputtering process took place, it caused the sputtered atoms spread out wide and not focused on the substrate surface. However, it is also identified that the increase of deposition time may increase the coverage area of the TiAlN layer.

From the SEM images show that TiAlN layer consists of a round and smooth micro particles. Increasing Al percentage affects the particle size become a much larger particles as presented in Table-3. In 10 % Al, the average diameter of TiAlN particles is 2.23  $\mu m$  which slightly increases to 3.39  $\mu m$  due to the addition of aluminium percentage in TiAlN composition. This phenomena caused by the presence of aluminium nitride (AlN) crystal growth inside the TiAlN thin layer.

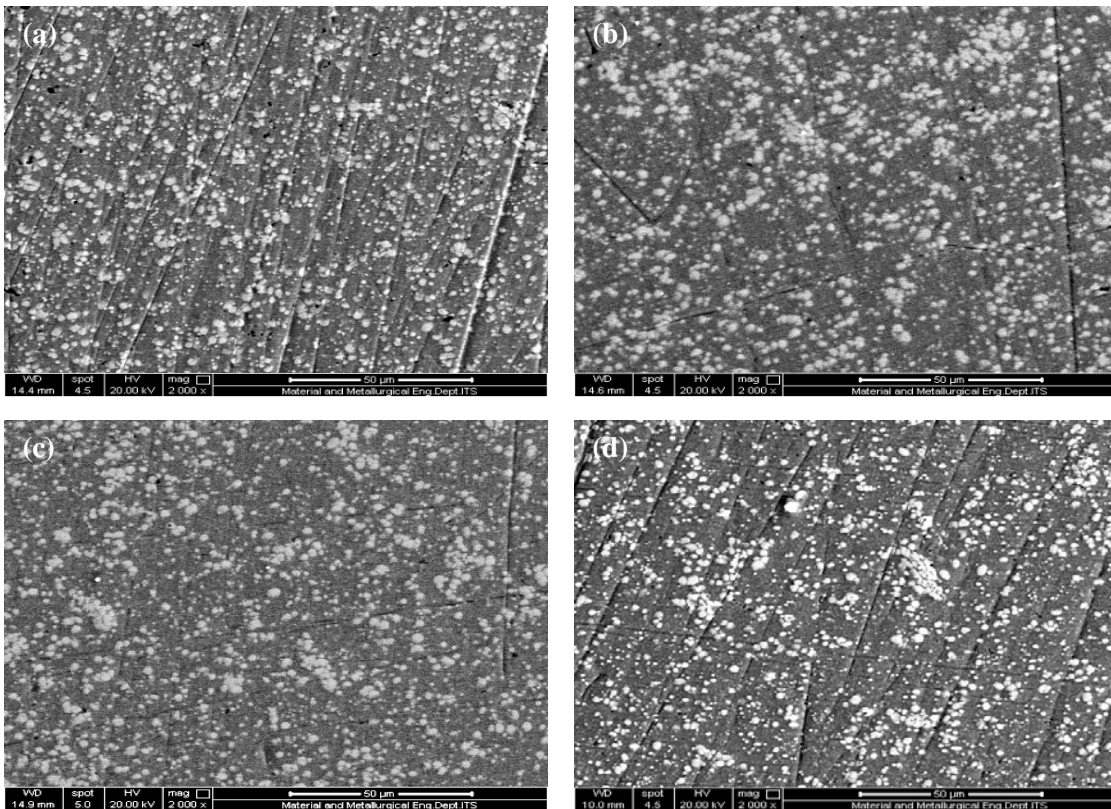
**Table-3.** Average particle size of TiAlN

Surface Ratio of Al (%)	Deposition Time (min)	Average Particle Size ( $\mu m$ )
10	15	2.23
	30	2.52
	45	3.04
20	15	3.06
	30	3.09
	45	3.10
30	15	2.73
	30	2.87
	45	3.08
40	15	2.81
	30	3.08
	45	3.39

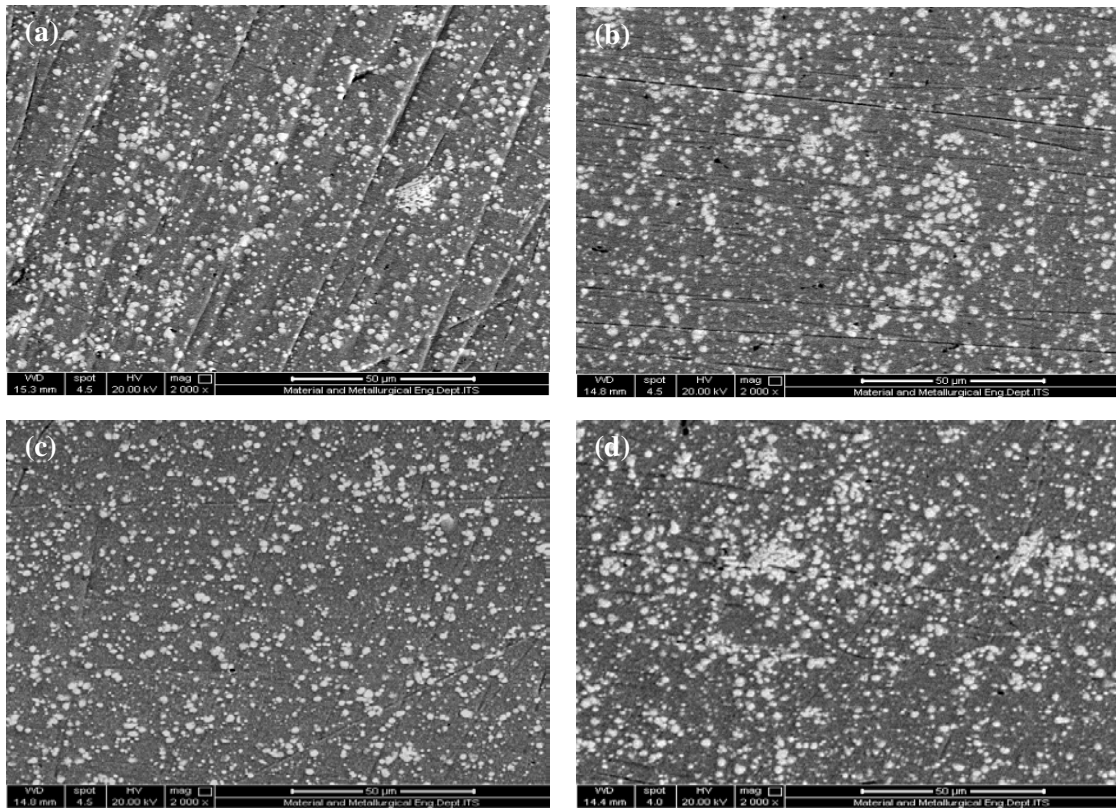
The surface topography images by AFM are shown in Figure-6 for an area of  $10 \times 10 \mu m^2$ . It is observed that increasing Al surface area ratio produces a much dense layer of TiAlN with homogeneous textures on the substrate material. While in the 10 % Al, a texture with a fewer number of peaks, lower and also narrower peaks are observed. From the topography images, it is also obtained information about surface roughness of TiAlN thin layer. The decline in surface roughness from 11.75 nm to 10.8 nm is observed when increased deposition time from 15 minutes to 45 minutes. The same thing occurs in 40 % Al, where in the deposition time of 15 minutes produces a layer with 12.8 nm surface roughness and then decreases to 12.6 nm with 45 minutes of deposition time.



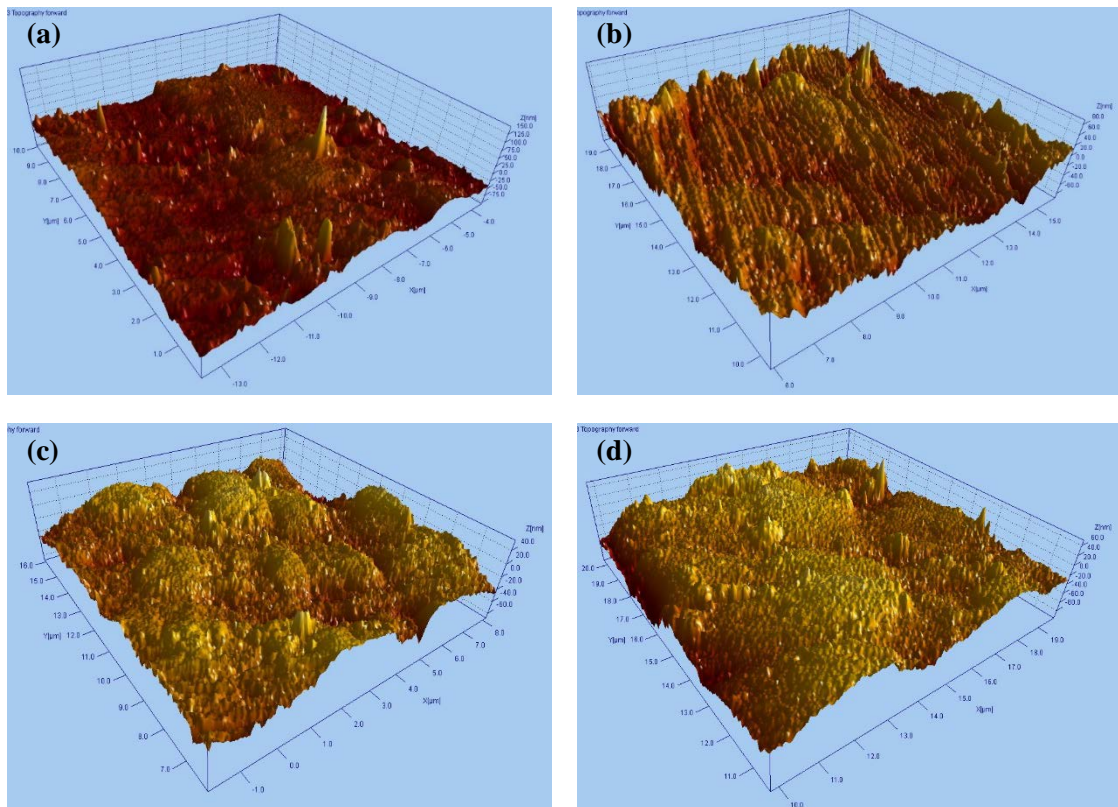
**Figure-3.** SEM images of the TiAlN surface with various surface area ratios of Al (a) 10 % Al; 15 min, (b) 20 % Al; 15 min, (c) 30 % Al; 15 min, (d) 40 % Al; 15 min



**Figure-4.** SEM images of the TiAlN surface with various surface area ratios of Al (a) 10 % Al; 30 min, (b) 20 % Al; 30 min, (c) 30 % Al; 30 min, (d) 40 % Al; 30 min



**Figure-5.** SEM images of the TiAlN surface with various surface area ratios of Al (a) 10 % Al; 45 min, (b) 20 % Al; 45 min, (c) 30 % Al; 45 min, (d) 40 % Al; 45 min



**Figure-6.** Topography images of TiAlN surface obtained by atomic force microscopy with various surface area ratios of Al (a) 10 % Al; 15 min, (b) 10 % Al; 45 min, (c) 40 % Al; 15 min, (d) 40 % Al; 45 min

#### IV. CONCLUSIONS

1. TiAlN thin layer were deposited on HSS AISI M41 by radio frequency sputtering. A mosaic target with various surface area ratios of Al to Ti obtained by put Al disks on the Ti target. This provides an easy and handy approach to fabrication of compound targets.
2. Increasing aluminium area on the sputtering target, produces TiAlN layer with a rich content of Al due to the sputtering yield of Al that much higher than Ti. While the deposition time does not significantly impact the TiAlN composition.
3. The thickness of TiAlN layer varies from 1.4  $\mu\text{m}$  to 5.2  $\mu\text{m}$ . The increase in thickness is attributed to the composition of TiAlN and also the deposition time. As the deposition time become longer, it increases the growth time of TiAlN layer on the substrate.
4. The structure of the TiAlN layers, was identified as a multiphase consist of cubic structure AlN and hexagonal TiAlN. With the addition of Al, the 2 $\theta$  angle slightly shift toward higher 2 $\theta$  value. Which caused by the stress release in the crystal that is characterized by impairment of micro strain in the crystal.
5. TiAlN layer consists of a round and smooth micro particles. Increasing Al percentage affects the particle size to become much larger particles due to the amount of AlN phase that grow in TiAlN layer.
6. 10 % Al with the deposition time of 45 minutes has a finest surface roughness of 10.8 nm. Increasing Al surface area ratios affect the surface topography of TiAlN layer to become much denser. While increasing deposition time affect the homogeneity of the TiAlN particles on the substrate.

#### V. ACKNOWLEDGMENTS

This research is supported by Department of Materials and Metallurgical Engineering, Institut Teknologi Sepuluh Nopember (ITS) Surabaya. The authors are also thankful to Pusat Sains dan Teknologi Akselerator Badan Tenaga Nuklir Nasional (PSTABATAN) for providing Radio Frequency Sputtering unit that used in this research and other facilities support.

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