Effect of Interlayer Coating Thickness on the Hardness and Adhesion for the Tungsten Carbide Cutting Tool

Kamil Jawad Kadhim Maher Ali Hussein Abbas Allawi Abbas
Al-Mussaib Technical Institute / Al-Furat Al Awsat Technical University
kamil_jawad35@yahoo.com maher.a.hussein81@gmail.com abbas_alawi80@yahoo.com

Abstract

The thin film of the (Al,Ti)N coating is studied with the aid of two parameters: hardness and adhesion. These parameters are very close to each other; however, in deposition field they could be interpreted differently. Several coatings of (Al,Ti)N layers are developed on tungsten carbide insert using the standard commercial $Al_{0.67}Ti_{0.33}$ cathodes in cathodic arc plating system(PVD). The influence of coating layer thickness on the mechanical properties of the coatings was investigated via two parameters: hardness and adhesion are characterized by the Rockwell tester Vickers tester. The measurements reveal that the highest hardness appears for the (Al,Ti)N thickness of 5.815 µm while the highest adhesion appears at a thickness of 3.089 µm. At the opposite extreme, the lowest hardness appears at 2.717 µm and the lowest hardness at 5.815 µm. Overall, the (Al/Ti) N coating of the thickness of 5.815 µm is controversial as it exhibits the highest hardness and the lowest adhesion. This result could be related to the effect of the formation of the micro-particle (MPs) which has a direct effect on the hardness because these MPs appear mainly on the surface and their presence at the interface is very limited. In addition, the creation of Ti buffering layer to reduce the delamination has its major effect on the adhesion but has no effect on the morphology of the surface. For these two reasons and the effect of the bias voltage, the results presented in this paper might show slight differences with other published papers. The composition of the (Al,Ti)N layer is characterized and, seemingly, it shows one important result which is showing that the ultimate composition of the (Al,Ti)N layer ($Ti_{0.62}Al_{0.38}$) is very close to the original target used in this study ($Al_{0.67}Ti_{0.33}$).

Keywords : Thickness layers, AlTiN coatings, Hardness, Adhesion.

الخلاصه

تم دراسة تأثير سمك الطبقة البينية لطلاء (Al,Ti)N على الخواص الميكانيكية للطلاء في عدة القطع باعتماد خاصيتي الصلادة والالتصاق .فقد تم طلاء عدة القطع باستخدام نترات الالمنيوم التيتانيوم (AlTi)N بنسب قياسية $Al_{0.67}Ti_{0.33}$ في نظام قوس كاثودي(PVD). وقد تم استخدام كل من اختبار روكويل وفكرز لقياس الصلادة والالتصاق. وقد اظهرت النتائج ان اعلى صلادة لطلاء (Al,Ti)N عند سمك 5.815µm بينما اعلى التصاق عند سمك 3.089µm وعلى العكس من ذلك فان اقل صلادة ظهرت عند 2.717µm في حين اقل التصاق ظهر عند 5.815µm. عموما فان طلاء (AlTi) Nعند سمك 5.815 يعتبر فعال اذ انه يعطي اعلى صلادة واقل التصاق ، وسبب انخفاض الالتصاق له علاقة بتأثير تشكيل الجسيمات الصغير(MPs) التي لها تأثير مباشر على الصلادة لان (MPs) تظهر اساسا على السطح ووجودها في الواجهة محدود جدا بالإضافة الى ذلك فان انشاء طبقة Ti اولية لتقليل الفصل بين الطبقات(التضمين) له تأثير كبير على الالتصاق ولكن ليس له تأثير على تركيب السطح. ولهذين السببين وتأثير جهد الحيز الكهربائي فان النتائج المعروضة في هذه الدراسة اظهرت اختلاف طفيف مع البحوث المنشورة الاخرى. ان تكوين طبقة (Al,TiN) تتميز على ما يبدو بإظهار واحدة من النتائج المهمة التي تبين التكوين النهائي لطبقات(Al,Ti N) بالنسب ($Al_{0.62}Ti_{0.38}$) قريبة جدا من الهدف الاساسي لهذه الدراسة ($Al_{0.67}Ti_{0.33}$).

الكلمات المفتاحية : سمك الطلاء، طلاء نترات الالمنيوم التيتانيوم ،الصلادة ، الالتصاق.

1. Introduction

Coating is relatively an old technique which has been rapidly developing in the last six decades (Abu-Shgair *et.al.*, 2010) due to its importance in manufacturing and the need for special materials for cutting tools. Coating could be can be carried out by several techniques such as magnetron sputtering, ion beam assisted deposition, arc evaporation and pulsed laser deposition (PLD). The basic purpose of coating is to protect surfaces of mechanical components working under high wear loads. The titanium (Ti) and aluminum (Al) are two elements played an important role in coating technology as they form a layer of (Ti, Al) or (Al, Ti) depending on the dominant material in the composition. The thickness of (Al, Ti) layer is crucial in some applications such as hardness, wear, and adhesion. Researchers are mostly interested in monolayer (Jakubéczyová *et.al.*,2012; Kottfer *et.al.*,2013; Abd Rahman,2009; Birol *et.al.*, 2012; Podgursky *et.al.*, 2011); multilayer (Jianxin *et.al.*, 2013; Ramadoss *et.al.*, 2013; Dejun *et.al.*, 2015; Kamil *et.al.*,2015;), and gradient layers (Chang and Wang, 2007). The thin Ti coating in particular is widely used for its good properties of improving adherence, hardness, reducing wear rate and coefficient of friction (Thornton et al.,1994; Grips et al.,2006;William and Retwisch,2010; Vamsi- Krishna *et.al.*,2010). A recent work by (Yong-Qiang *et.al.*,2011) suggested that TiN/TiAlN and TiN/AlTiN multilayer coatings deposited on cutting tools could improve the mechanical and corrosion resistance.

It has been shown that (Al,Ti)N coating has better mechanical properties compared to TiN due to the inclusion of aluminum atoms in TiN crystalline structure which results in increasing oxidation resistance by formation of the stable ternary material (Al,Ti)N (Verma *et.al.*2012; Cselle *et.al.*,2009). Recently, a new coating technology known as pulsed bias arc ion plating (PBAIP) which has advantages over other technologies due to its lower processing temperature requirement, lower residual stress, better grain refinement and particle purification (Soediono,1989; Bunshah, 1994; PalDey *et.al.*, 2003).PBAIP also provides the ideal conditions for the deposition of a multilayer film with excellent performance. Titanium Aluminum Nitride (Ti,Al)N or (Al,Ti)N, as in this work, is suitable for coating cutting tools for machining hard to cut materials because the coating can withstand extreme environments such as high temperature and high pressure condition. These properties made TiAIN suitable for use in dry machining and high-speed milling and turning where heat is generated (Chou *et.al.*,2002; Siow *et.al.*,2013; Li *et.al.*,2013;Feng *et.al.*, 2013).When properly applied, it enhances the cutting speed and the feed rate, ultimately improving productivity. The coatings are known to be relatively hard coatings with superior oxidation resistance, chemical inertness and good thermodynamic stability (Richard *et.al.*1992;Padley and Deevi,2003). An atomistic deposition process can be performed in vacuum, plasma, or gaseous.

The mechanical properties of thin film (Al,Ti)N is studied using different techniques by determining certain parameters. In this present work, two parameters are determined: the hardness and adhesion. The hardness is defined as the resistance of a material to scratching. Hardness is explored by the microscopic behavior of the material which characterized by the strength of the molecular bonds. Hardness is normally measured by a normal scratch, indentation scratch. The hardness is measured by a unit associated with Vickers Hardness machine which is abbreviated by HV where 1 MPa is equivalent to 9.807 HV. Adhesion, on the other hand, is characterized by the chemical and physical bonds between two adjacent materials and it could be determined by the force required to separate the two surfaces. Adhesion depends on three factors: bonding across the interface, the type of the interface, and the fracture mechanism which causes failure.

The hardness and the adhesion are very sensitive to the coating technique which results in the presence of macro-particles. In this paper, the hardness and adhesion are analyzed in conjunction with the physical uniqueness of these findings plays an important role in determining the durability and the usability of the thin film.

2. Experimental coating plant

The coating experiments were conducted using the arc ion plating system made by J&L Technology, Korea, model Legend H.I.P.III. Process gasses used are Argon (Ar) gas MFC capacity 1000 SCCM and Nitrogen (N_2) gas MFC capacity 1000 SCCM. The Arc power supply (10KW, 50V, 200A) and the bias power supply

(18KW, 1200V/15A). Al0.67Ti0.33 of 99.99% purity and Ti were used as targets with diameter and thickness of 150 mm and 15 mm, respectively. The substrate jig holder (turntable) has 6 axes with triple rotations. The thermocouples were located at the back of the substrate jig holder with high, mid, and low level from top to bottom. They were approximately 20-30 mm from the substrate holder. The intended AlTiN coating were deposited on the tungsten carbide insert for substrate in the presence of the nitrogen gas inside the coating chamber. This system is a metal cathodic arc ion plating system utilizing arc discharge for hard thin film coating. The device generates plasma through the metal arc sources, and is available for various kinds of thin film coating such as AlTiN and TiN. The substrate is a WC square cutting insert dimension is 12.7 x 12.7 x 3.18 mm.

3. (Al,Ti)N coating deposition

Prior to coating, the cutting insert was cleaned by ultrasonic with a detergent bath for different mixed detergent and time as shown in Table 1: using ultrasonic system. The cleaning procedure consists of six steps through which the cutting sample under investigation is clean from any impurities which could affect its physical and mechanical properties.

Table 1: Cleaning Procedure

Tank #	Solvent	Time (s)	Temperature ℃	Condition
1	Deconex, DIwater: Corrosion	300	52	Rotation
2	Deconex, DI water: For ultrasonic	300	52	Rotation
3	DI water			Spray/Rotation
4	Deconex,DI water: For carbide and HSS	150	52	Rotation
5	DI water			Ultrasoic/Rotation
6	DI water: Rinsing			Rotation

The parameters setting for main AlTiN coating layer deposition processes in Table 2. The parameters include rotation speed, time allocated for deposition, the type of gas used, the bias voltage and the substrate temperature.

Table 2: Deposition parameters

Rotational Speed (rpm)	AlTiN Disposition Time (min)	Total Layer Thickness μm	Argon Flow Rate (sccm)	N_2 Gas Flow Rate (sccm)	Bias Voltage (-Volt)	Buffer layer (min)	Etching Time (min)	Substrate Temp ℃
5	45	2.717	100	600	40	10	30	380
5	80	3.089	100	600	40	10	30	380
5	100	3.912	100	600	40	10	30	380
5	120	5.815	100	600	40	10	30	380
5	135	8.760	100	600	40	10	30	380

The deposition system which is utilized in this work is schematically shown in Figure1.

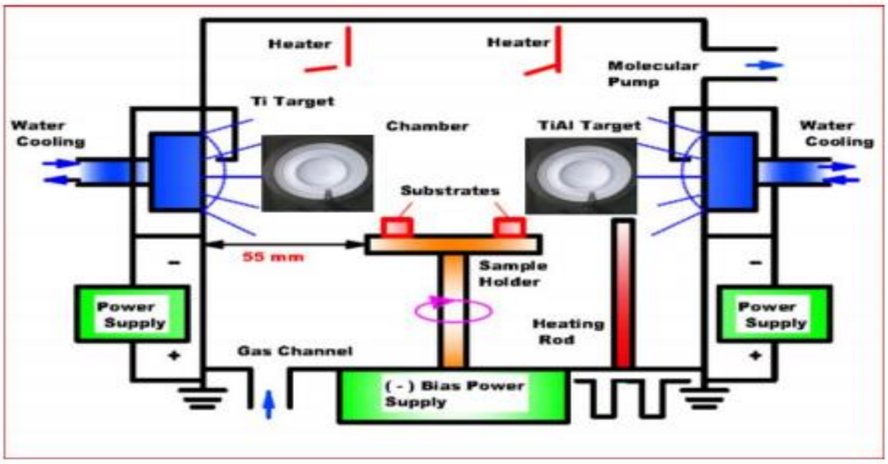


Figure 1: Schematic diagram of the arc ion plating system

4. Micro hardness

The micro-hardness was tested using Vickers micro hardness testing machine (Vickers tester, VLPAK-2000 Mitutoyo Microwizhard). A 50 gm loading is used to test the hardness. Experimentally, five measurements were taken for each specimen and the average values were determined.

5. Adhesion

The adhesion between AlTiN coating and WC substrates was determined using Rockwell indenter (OMAG 250-3302MRS) with a Brale diamond indenter an applied load of 588, 980, and 1470 N (Wu et al., 2000). The boundary of the crack was determined based on visual inspection through the captured image from optical microscope based on the delamination of the coating layer and the formation of the crack. The adhesion properties were determined by analyzing the crack formation after the indentation. A typical measurement of crack diameter vs. applied load is conducted. The lateral crack diameter of indentation was plotted against the three different applied loads and a best fit line was generated based on the three points. The slope of the line represents the adhesion strength of coatings.

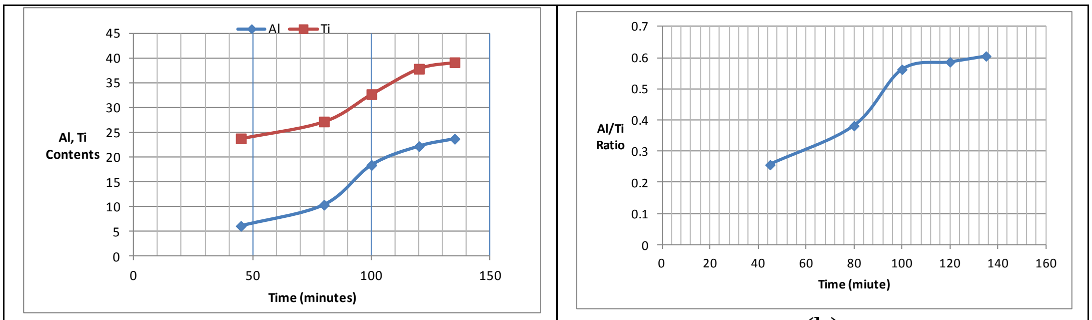
6. Results and Discussion

Five samples are prepared by a series of steps started with a special procedure of cleaning the substrate as proposed in Table 1. The parameters used for performing coating process are listed in Table 2. The coating of (Ti, Al) N five samples is prepared throughout the system shown in Figure 1. The five samples are investigated using SEM/EDX for thickness and morphology [R]. The results which are related to the present paper are tabulated in Table 3.

Table 3: Analysis of as-deposited (Al,Ti)N coatings

Run	Coating time (min)	Thicknes s µm	Al	Ti	Total Al and Ti	Al/Ti %	x: Ti	1-x: Al	Ti_xAl_{1-x}
1	45	2.717	6.09	23.75	29.84	26	0.79	0.21	$Ti_{0.79}Al_{0.21}$
2	80	3.089	10.36	27.16	37.52	38	0.72	0.28	$Ti_{0.72}Al_{0.28}$
3	100	3.912	18.38	32.68	51.06	56	0.64	0.36	$Ti_{0.64}Al_{0.36}$
4	120	5.815	22.17	37.82	59.99	58	0.63	0.37	$Ti_{0.63}Al_{0.37}$
5	135	8.760	23.67	39.67	62.79	61	0.62	0.38	$Ti_{0.62}Al_{0.38}$

It’s important here to present one important factor which is the percentage ratio of Al/Ti. The content of Al and Ti in each sample which was characterized by relevant techniques [R] are plotted in Figure 2a , while the ratios of Al/Ti are shown in Figure 2b.

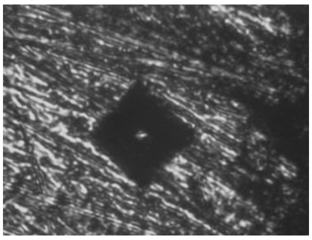


(a) (b)

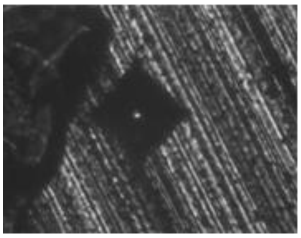
Figure 2. (a) The content of Al and Ti in (Ti,Al)N samples, (b) Al/Ti ratio.

7. Micro-hardness Analysis

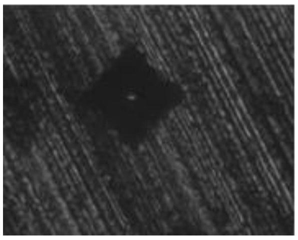
The five samples of coating (Al,Ti)N of thickness 2.717 μm to 8.760 μm are shown in Figure 3 (a through e).The hardness is tested by the Vickers micro hardness tester (VLPAK-2000 Mitutoyo micro wizhard) with applied loads of 50 g. For each specimen, the average of hardness value was taken at least from five test readings.



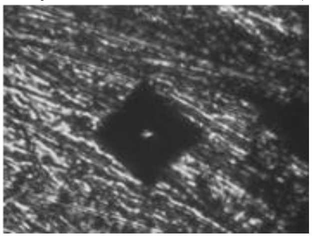
(a) 2.717µm



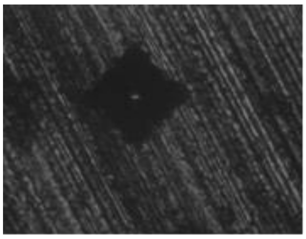
(b) 3.0818µm



(c) 3.912µm



(d) 5.815µm



(e) 8.760µm

Figure 3: Hardness of AlTiN coating load 50g and indenter tip. Micro-hardness indentation test, (mag = 1.000 kx).

Table 4 shows the average values of the hardness of each sample of specified thickness taken at five arbitrary locations X_1 through X_5 using a 50-g load. The hardness was found to be 1692.8 HV at Al/Ti ratio of 26%. As Al/Ti ratio increases to 38%, the hardness increases and then decreases when the Al/Ti ratio approached the 56% level. The results are deviated from those proposed by Paldey et al. (Paldey et al., 2004) who reported that the hardness decreases when the Al fraction is higher than 30%. The results here show that the hardness increases from 1692.8 HV at Al/Ti ratio of 26% to 1811.2 HV at a ratio of 38% in agreement with the findings of Paldey et al., 2004. However, as the Al/Ti ratio increases to 56%, the hardness decreases in almost lowest value ,the hardness go down at this point because microparticle size very large . The hardness then increases before showing a slight decrease in the Al/Ti ratio of 61%. The trend of the hardness behavior in this work has deviated from Paldey and Deevi's work for a number of reasons. One possible reason is due to the different conditions and parameters of conducting the coating especially in the buffering Ti layer which was set in this work after 10 minutes, but was not mentioned in Paldey et al., 2004 work. The other possible reason is that the formation of MPs which has a severe impact on some parts of the surface , especially those parts were hit by ionized particle which are driven by the bias voltage (Wei and Gong, 2011;

Yong-Qiang et al., 2011; Martinez et al., 2014). Figure 4 shows the variation of the hardness of the (Ti,Al)N surface as a function of coating thickness. As indicated in Figure 4 and shown in Table 4, the hardness go down at thickness 3.089 µm because microparticle size very large at this thickness , the maximum hardness was achieved at a coating thickness of 5.815 µm where after this achievement, the hardness decreases.

Table 4: Micro-Vickers hardness at different thickness of coated (Al,Ti)N.

Thickness µm	X_1 HV_{50g}	X_2 HV_{50g}	X_3 HV_{50g}	X_4 HV_{50g}	X_5 HV_{50g}	X_{avg} HV_{50g}	Al/Ti%	Ti_xAl_{1-x}
2.717	1680	1641	1780	1700	1663	1692.8	26	$Ti_{0.79}Al_{0.21}$
3.089	1992	1743	1667	1940	1714	1811.2	38	$Ti_{0.72}Al_{0.28}$
3.912	1687	1765	1684	1684	1733	1703.0	56	$Ti_{0.64}Al_{0.36}$
5.815	1808	2098	2080	2080	1912	1939.0	58	$Ti_{0.63}Al_{0.37}$
8.760	1851	1761	1851	2062	1851	1875.0	61	$Ti_{0.62}Al_{0.38}$

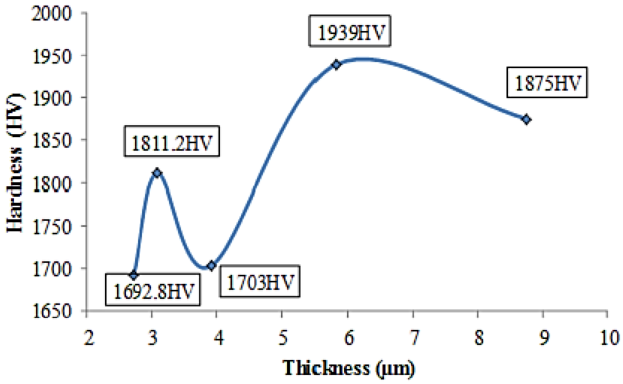


Figure 4: Hardness of AlTiN layer coatings with different thickness and deposition time.

The fluctuation of the hardness of (Ti,Al)N samples shown in Figure 4 could be interpreted using other approaches. The higher level of arcing of Al compared to Ti during deposition of ions onto the growing film due lower atomic mass of Al has severe impact on the hardness of the coating (Nizam, 2009; Zhao *et.al.,* 2012). In addition, the ratio of Al/Ti which is shown in Figure 2b is not linear which means the deposition rate of Al or Ti is different from one another. The coating composition could be attributed to higher levels of arcing of Al compared to Ti during deposition onto the growing film due lower atomic mass of Al compared to Ti atoms [1, 12]. In addition, the irregularity in Al/Ii ratio as thickness increases could be attributed to either higher Al deposition tendency over Ti tendency due to their atomic size, or due to the influence of N which is very compatible to the Al rather than the Ti, or due to the variation of ionization grade of Ti and Al [1, 2]. In addition, as Al/Ti ratio increases, the film hardness decreases and reaches a value of 1875.0 HV at coating thickness of Al/Ti at 5.815 µm where the Al/Ti ratio is 58%.

8. Coating Adhesion Analysis

The adhesion between (Ti,Al)N coating and WC substrate is determined using Rockwell indenter (OMAG 250-3302MRS) with a Brale diamond indenter at applied loads of 588, 980 and 1470 N. The coating adhesion was analyzed through the adhesion slope measurement method. The adhesion slope is calculated through the analysis of crack formation after creating indentation using optical microscope with 20X-magnification. The lateral crack diameters of the indentation for three different applied loads (588 N, 980 N, and 1470 N) are determined and the values are tabulated in Table 5.

Table 5: Crack diameter and adhesion slope for various coating thickness.

Thickness (μm)	HRA/588 (μm)	HRA/980 (μm)	HRA/1470 (μm)	Adhesion Slope
2.717	100.82	160.02	127.14	0.0671
3.089	101.93	160.03	131.52	0.0655
3.912	105.22	161.11	134.81	0.1824
5.815	100.83	293.73	165.50	0.2205
8.760	173.17	309.07	232.35	0.1542

The data of the adhesion slopes in Table 5 are plotted in Figure 5. The slope of this line indicates the adhesion strength of the coating. The lower the slope, the better the adhesion strength is (Wu. *et.al.*, 2000). At the first two thickness values of 2.717 μm and 3.089 μm, the adhesion slopes are low suggesting that the adhesion is the highest. When the thickness increases to 3.912 μm, the adhesion strength decreases by two-third showing the least adhesive force at a thickness of 5.815 μm. There is a slight relief in the adhesion force when the thickness increases to 8.760 μm.

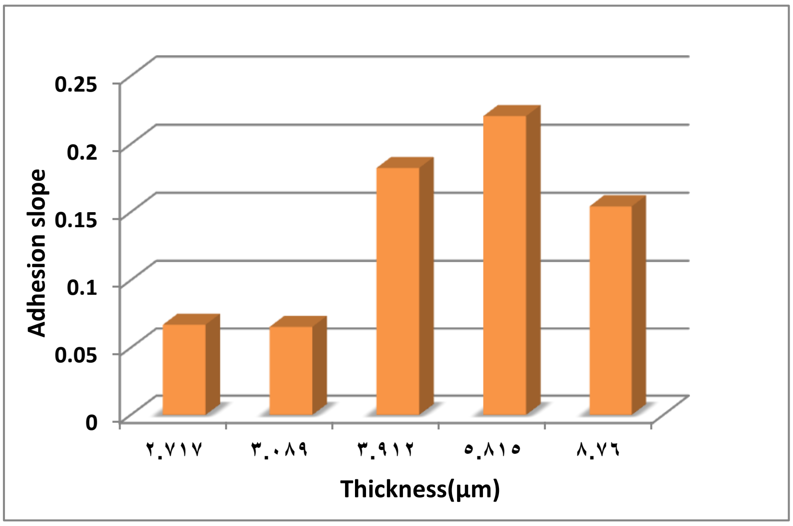
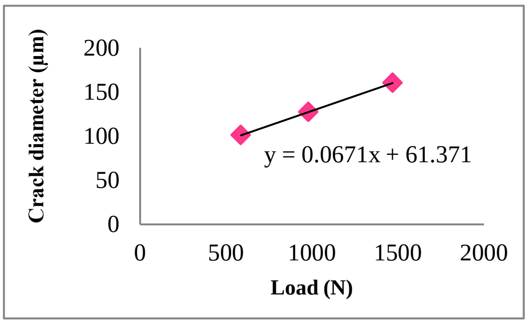
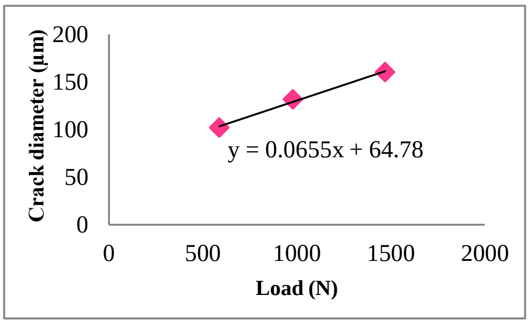


Figure 5: Adhesion slope vs thickness

It is very important and for sake completeness, the adhesion of (Al,Ti)N is analyzed in terms of load used in the test as shown in Figure 6. In this analysis, the crack formation after creating indentation is utilized and tested by an optical a 20X-magnification microscope. The relation between the crack diameter and the load are linear which is expected. The highest adhesion strength, corresponds to lower slope of the straight line (Figure 6b), was achieved for (Al,Ti)N coating thickness of 3.089 μm at Al/Ti% ratio of 38% and the structure of the thin film is $Ti_{0.72}Al_{0.28}$.



(a)



(b)

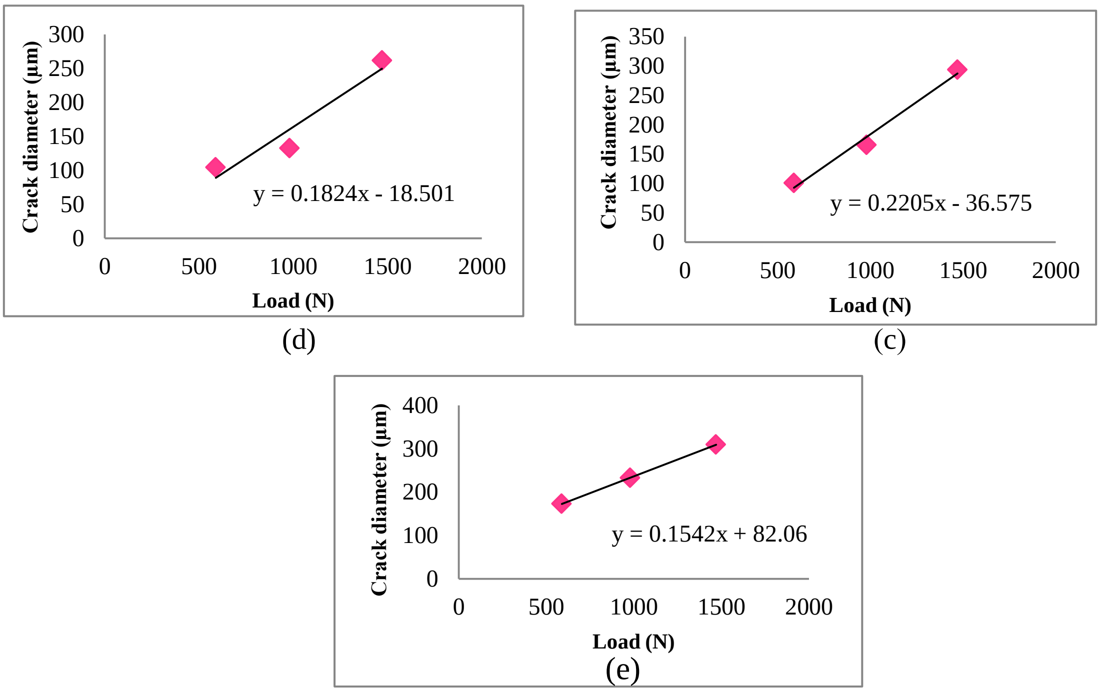


Figure 6: Adhesion Load of (Al,Ti)N coatings with different layer thickness: (a) 2.717µm,(b) 3.089µm, (c) 3.912µm, (d) 5.815µm, (e) 8.7608µm.

9. Conclusions

The thin film of the (Al,Ti)N coating is studied with the aid of two parameters: hardness and adhesion. These parameters are very close to each other; however, in deposition field they could be interpreted differently. The hardness reveals that the best coating corresponding to the highest hardness appears in the thin film of 5.815 µm where the Al/Ti ratio is 58% and the film characterized by $Ti_{0.63}Al_{0.37}$. On the contrary the lowest hardness appears at 2.717 µm-thicknesses, where the Al/Ti ratio is 26% and the characterization of the film is $Ti_{079}Al_{0.21}$. The adhesion measurements suggest that the highest adhesion corresponds to 3.089 µm, Ti/Al ratio of 38%, and film characterized at $Ti_{079}Al_{0.21}$. The lowest adhesion appears for the 5.815 µm, al/Ti ratio of 58% and $Ti_{0.63}Al_{0.37}$.The different apparent results of the hardness and the adhesion could be related to the formation of the macro particles (MPs) which appear normally on the surface and it has a direct impact on the hardness but it has, to a lesser extent, the effect on the adhesion since the adhesion is concerned about the mutual adjacent surfaces. Other factors could also play a role, such as the parameters under which the thin film is developed.

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