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ADHESION OF THIN COATING ON ALUMINUM ALLOYS

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ABSTRACT

ADHESION OF THIN COATING ON ALUMINUM ALLOYS. Adhesion of thin coating on aluminum alloy was studied. Adhesion of thin coating on a substrate is a key parameter in surface engineering. Adhesion testing becomes interesting thing to be applied in characterization of a coating, even though the fundamental understanding of the process is still limited. Adhesion evaluation of a coating can be carried out using many methods and one of them is scratch method. Scratch was performed on a surface of coating by means of an indenter. The indenter should be very hard such as a diamond. Scratch was obtained by moving the sample under indenter slowly with a constant speed. A progressive increase or stepwise load was given. The scratches were evaluated by means of a scanning electron microscope. In this study, adhesion of metallic coating-copper, nickel, chromium and ceramic coating-titanium nitride, chromium nitride on aluminum alloy and that on nickel deposited aluminum alloy were evaluated. Scratch testing with a stepwise load was applied. Three parameters of adhesion-critical load, adhesion index and interface toughness were discussed. The results show that scratch testing is suitable for evaluating the above coating. Adhesion of duplex coating is higher than that of single coating and adhesion of duplex nickel coating is the highest.

Key words : Adhesion, thin coating, aluminum alloys

ABSTRAK

ADHESI LAPISAN TIPIS PADA PADUAN ALUMINIUM. Adhesi lapisan tipis pada paduan aluminium telah diteliti. Adhesi lapisan tipis pada suatu substrat merupakan satu parameter penting dalam perekayasaan permukaan. Uji adhesi menjadi menarik untuk digunakan mengkarakterisasi suatu lapisan, walaupun pemahaman proses yang terjadi masih terbatas. Evaluasi adhesi suatu lapisan dapat dilakukan dengan berbagai cara dan satu diantaranya adalah metode goresan. Goresan dibuat pada permukaan lapisan menggunakan indenter. Indenter yang digunakan harus keras seperti intan. Goresan didapat dengan menggerakan sampel dengan perlahan dan kecepatan tetap. Indenter diberi beban yang terus meningkat atau tetap bertahap. Goresan dievaluasi dengan menggunakan *Scanning Electron Microscope*. Dalam penelitian ini adhesi lapisan logam-tembaga, nikel, kromium dan lapisan keramik-titanium nitrida, kromium nitrida pada paduan aluminium dan pada paduan aluminium yang terlapis nikel dievaluasi. Uji goresan dilakukan dengan menggunakan beban tetap bertahap. Tiga parameter adhesi-beban kritis, indeks adhesi dan ketangguhan antar muka didiskusikan. Hasil pengujian memperlihatkan bahwa uji goresan dapat digunakan untuk mengevaluasi lapisan-lapisan tersebut diatas. Adhesi lapisan duplek lebih tinggi dibandingkan dengan adhesi lapisan bukan duplek dan adhesi lapisan nikel duplek adalah yang tertinggi.

Kata kunci : Adhesi, lapisan tipis, paduan aluminium

INTRODUCTION

Due to many special properties of this alloys, such as light weight high thermal conductivity, high corrosion resistance, aluminum is an attractive alloy for engineering application. However, since their poor wear resistance, their usefulness in many engineering application is impaired [1]. A large amount of research works has been carried out to enhance the properties of this alloy. One of the techniques improving the above property is coating. Aluminum alloys can be coated with innovative processes such as physical vapor deposition [2]. This technique provides an opportunity to coat aluminum alloy with metal or ceramic. A coating on a substrate that has already been coated with other materials or other technique is called a duplex coating.

In order to keep high wear resistance, coating should have good adhesion to substrate. Adhesion of a coating to substrate can be measured using several methods and one of them is scratch method. Scratch is made on the surface of coated specimen with a certain load and speed. Critical load as determined by scratch test is often used to express adhesion between coating and substrate. Critical load changes with thickness of coating. The thicker the coating, the higher the critical load. Adhesion index is also used to represent adhesion of coating to substrate. This parameter is similar to critical load which changes with coating thickness. Interface toughness can also be used to illustrate adhesion of coating to substrate.

In this study, adhesion of metallic coating-copper, nickel, chromium and ceramic coating - titanium nitride, chromium nitride on aluminum alloy were evaluated in addition to adhesion of the above coating on nickel deposited aluminum alloy. The adhesion of coating to substrate is expressed as critical load, adhesion index and interface toughness.

EXPERIMENTAL METHOD

Specimen used in this study is aluminum alloy containing 10.7 % Si-1.2 % Mg-1.0 % Cu-0.4 % Fe-Al balance. Specimens were aged at 180 ^oC for 4 hours prior to coating. Some specimens were coated directly while the others were nickel deposited first before coating.

Copper coating was carried out by means of physical vapor deposition technique. A tungsten boat was used for evaporation. Melting and evaporation of copper started after sputtering for 30 minutes in an argon atmosphere. The melt was stirred and bubbled until it was totally evaporated. The coating is performed to obtain 3 micrometer thickness. The substrate temperature during coating was around 200 ^oC.

Chromium coating was also carried out by means of physical vapor deposition technique. A tungsten boat was used for evaporation of chromium. Chromium was sublimed instead of melted. The coating is performed to obtain 3 micrometer thickness. The substrate temperature during coating was around 240 ^oC.

Nickel coating was carried out by means of physical vapor deposition system. An electron beam heating source was used to melt nickel. A water cooled copper boat was used instead of a tungsten boat, since the tungsten boat will react with nickel at elevated temperature. The coating is performed to obtain 3 micrometer thickness. The substrate temperature during coating was around 250 ^oC.

Titanium nitride and chromium nitride coating were carried out for both type of specimen in the same way as metal coating above. Coating was deposited to a thickness of 3 micrometer. The substrate temperature was constant during coating.

Scratch testing were done using Leitz microhardness tester with a 120 degree prism diamond indenter. One of prism diagonal is parallel to scratch direction. Scratch is performed at a various load from 5-500 grams by moving the specimen under indenter slowly with a constant speed of 0.05 mm/second. The scratches were analyzed using a scanning electron microscope.

RESULTS AND DISCUSSION

Scratches on duplex nickel coating specimen with a various load, from 10 to 100 grams are presented in Figure 1. This figure shows that scratch width increases with increasing load. This figure also show that the coating fail at the load of 100 grams.

Scratch on duplex CrN coating specimen with a load of 50 grams is shown in Figure 2. This figure clearly shows that the load used in current experiment does not

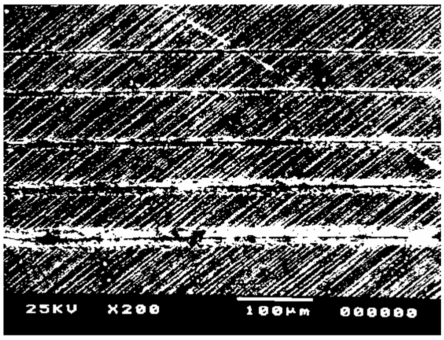

Figure 1. Scratches on duplex nickel coating specimen with a various load, from 10 to 100 grams (top to bottom).

exceed the critical load of the specimen, the load that produced coating damage. No coating damage was observed.

Figure 3 shows the scratch on duplex CrN coating specimen with a load of 100 grams. This figure shows that the load used in current

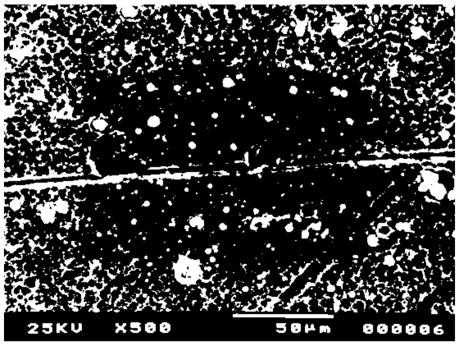

Figure 2. Scratch on duplex CrN coating specimen with a load of 50 grams and a speed of 0.05 mm/second.

experiment exceed the critical load of the specimen. Coating damage was observed. Based on both observation, the critical load of duplex CrN specimen is $50 < L_c$ d” 100.

Based on direct observation using a scanning electron microscope, the critical load for all coating is presented in Table 1.

In addition to direct observation of adhesion of thin coating using a scanning electron microscope, indirect observation is carried out. In this observation, scratch width is measured for a given load.

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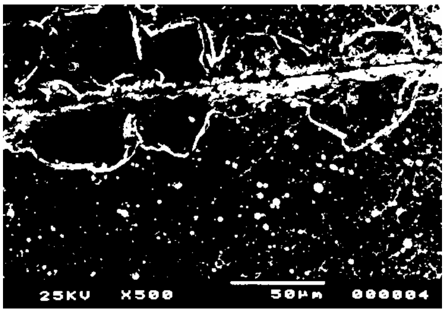

Figure 3. Scratch on duplex CrN coating specimen with a load of 100 grams and a speed of 0.05 mm/second.

Table 1. Critical load for the coating.

Coating	Critical Load (gram)
Cu	$15 < L_c \leq 25$
Ni	$25 < L_c \leq 50$
Cr	$25 < L_c \leq 50$
TiN	$15 < L_c \leq 25$
CrN	$25 < L_c \leq 50$
Duplex Cu	$25 < L_c \leq 50$
Duplex Ni	$50 < L_c \leq 100$
Duplex Cr	$50 < L_c \leq 100$
Duplex TiN	$25 < L_c \leq 50$
Duplex CrN	$50 < L_c \leq 100$

Observation data for the above experiment is presented in Tabel 2.

It is reported [3,4] that for a thin coating, scratch width obtained from scratch testing follows the equation

$$L = K_1 d^2 \quad \text{.....} \quad (1)$$

Where L is a given load, d is scratch width and K_1 is a constant which depends on elastic energy of coating, Young modulus of coating, coating thickness, friction coefficient of coating and indenter geometry. The above equation can be written as:

$$L^{½} = K\,d \quad \text{.....} \quad (2)$$

Where K is square root of K_1. Every thin coating has two values of K. First, for lighter load, the stylus is in the coating and scratch does not damage the thin coating. Value of K is dominated by the characteristic of thin coating. Second, for heavier load, stylus is in the substrate and scratch damage thin coating. The value of K is dominated by the characteristic of substrate. Figure 4 to 8 show $L^{½}$ vs d graphs obtained from the above experiments.

It is also reported [5,6] that for a thin coating, scratch width obtained from scratch testing follows the equation:

$$L = K_2\, d^2 / t^{½} \quad \text{.....} \quad (3)$$

Where K_2 is a constant which depends on elastic energy of coating, Young modulus of coating, friction coefficient of coating and indenter geometry. It does not depend on coating thickness.

Critical Load

As mention before that there are two types of observations on adhesion of thin coating: direct and indirect. Direct observation is based on coating damage produced during scratch experiment. The critical load of this observation is not an exact value but a range between two adjacent loads that produced coating damage and not. On the other hand, indirect observation is based on scratch width produced during scratch experiment. There are two methods to find a critical load, intercept and critical scratch width methods.

Based on scratch width and load, graph of load versus scratch width square is made to be used to find a linier regression line for heavier load. The intercept of the linier regression line and load axis is the critical load. It is understood, if there is no coating, the intercept will be at the origin (0,0) while if there is a coating, the intercept between regression line and the load axis will be at (0,c). Therefore, c is the critical load of coating.

Similar to intercept method, the following method is based on scratch width and load. A graph of load versus scratch width square should be obtained. Critical scratch width is defined as scratch width when the tip of indenter reaches the coating-substrate interface. Critical load is obtained if a graph of load versus scratch width square and critical scratch width is available. It is

Table 2. Scratch width for a given load.

Load (gram)	Scratch width (□m)									
	Cu	Ni	Cr	TiN	CrN	d-Cu	d-Ni	d-Cr	d-TiN	d-CrN
5	2	1	1	2	1	1	1	1	1	1
10	4	2	2	4	2	2	2	2	2	1
15	8	4	3	6	3	6	3	3	4	2
25	15	7	7	10	6	10	5	5	7	4
50	25	15	15	20	13	17	10	10	13	8
100	50	30	30	40	28	30	20	20	25	17
200	75	60	60	70	55	55	40	38	50	35
300	100	85	85	95	82	80	60	6	75	55
500	145	125	125	135	125	120	100	100	105	90

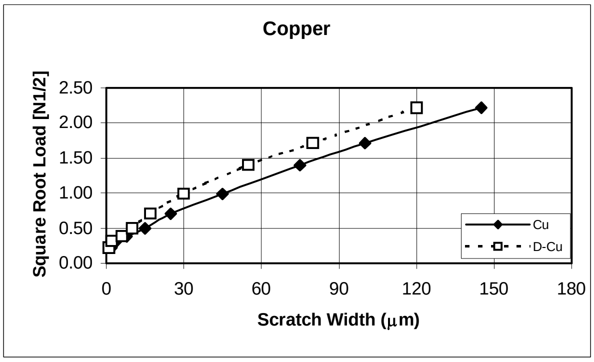

Figure 4. $L^{½}$ versus d graphs of Cu coating and duplex Cu coating on aluminum.

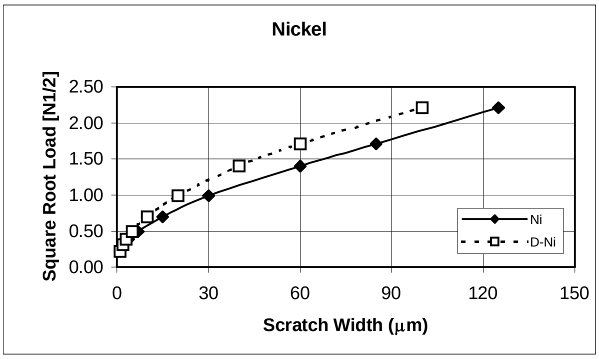

Figure 5. $L^{½}$ versus d graphs of Ni coating and duplex Ni coating on aluminum.

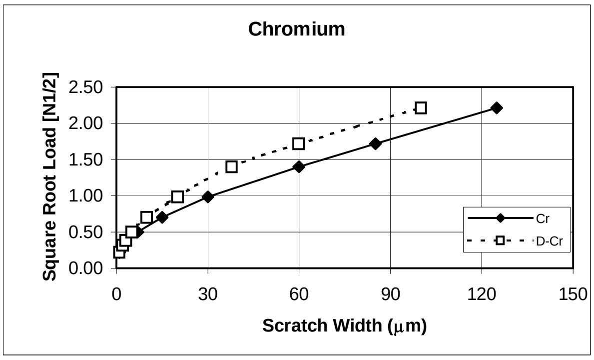

Figure 6. $L^{½}$ versus d graphs of Cr coating and duplex Cr coating on aluminum.

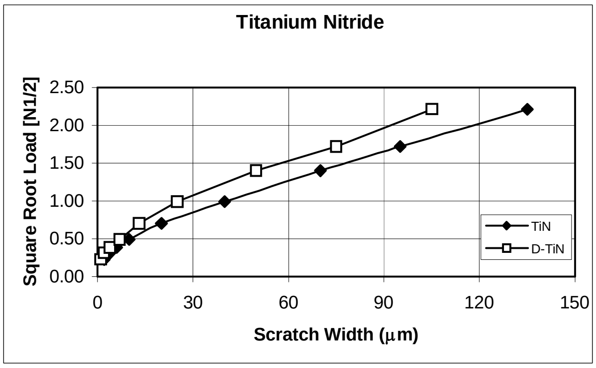

Figure 7. $L^{½}$ versus d graphs of TiN coating and duplex TiN coating on aluminum.

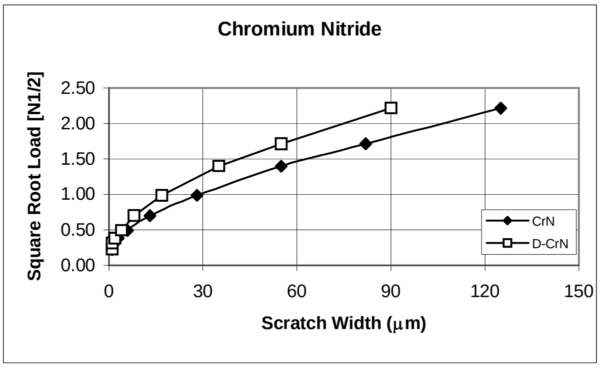

Figure 8. $L^{½}$ versus d graphs of CrN coating and duplex CrN coating on aluminum.

understood if the indenter is in the substrate the interaction between indenter and substrate can not be ignored. Therefore, if critical scratch width is known and the graph of load versus scratch width square is available the critical of the coating is obtained.

For thin coating with t thickness and 120 degree prism indenter, critical scratch width will be $2t\sqrt{3}$ Using the graphs shown in Figure 4-8 and critical scratch width for each specimen, the critical load is obtained. The critical load is shown in Table 3

The three methods of measuring critical load have their advantage and disadvantages. The advantage of direct method is that the critical load can be obtained directly and the disadvantage is that the critical load is not an exact value. The critical load lies in a range between two adjacent load that produces coating damage.

On the other side, advantage of intercept method is critical load which is obtained in an exact value. Applying this method on thin hard coating on soft substrate should be done carefully. Data needed for this method is obtained at heavy load. For a heavier load, thin hard coating on soft substrate is usually broken into pieces. The last method is scratch width method. This method provides an exact value of critical load, but to find the critical load, thickness of the coating should be known.

It is reported [5] that critical load of 2 μm titanium nitride on tool steel is about 16,3 gram, while the critical load of 3 μm titanium nitride on aluminum alloy in this study is about 23,9 grams. The different between the two results can be understood since critical load is thickness dependent. The thicker the coating the higher the critical load.

Critical load of duplex coating is higher that that of single coating. It is caused by the presence of intermediate coating. This intermediate coating acts as thin coating, therefore, the elastic energy stored in the coating increases and the coating damage will occurr at a higher load.

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Adhesion Index

Adhesion index is defined as slope of load versus scratch width square graph at the critical load. Adhesion index cannot be observed directly. It is only possible by measuring the scratch width and the correspond the load. In addition, this index cannot be obtained unless the critical load is available. From equation 1, it is obviously seen that K_1 is the adhesion index if L is the critical load that corresponds with d which is the scratch width.

Adhesion index for specimen observed is presented in Table 4. It is clearly seen that adhesion index for single coating is lower than that for duplex coating. Duplex nickel coating has the highest adhesion index. Adhesion index of duplex chromium nitride coating is higher than that of duplex titanium nitride coating. Adhesion index of copper coating is the lowest.

Table 4. Adhesion Index and Interface Toughness..

Coating	Adhesion Index (10^9 Nm^{-2})	Interface Toughness (10^6 $Nm^{-3/2}$)
Cu	2.657	3.068
Ni	5.244	6.056
Cr	5.451	6.294
TiN	3.317	3.831
CrN	6.426	7.420
Duplex Cu	5.280	6.097
Duplex Ni	8.491	9.805
Duplex Cr	8.190	9.458
Duplex TiN	5.309	6.131
Duplex CrN	8.345	9.636

Interface Toughness

This is the third parameter that is used to represent adhesion of a coating to a substrate. The first two parameters are critical load and adhesion index. These parameters change with thickness coating. The thicker the coating is the higher the critical load and the higher the adhesion index.

Interface toughness is defined as slope of load versus $d^2/t^{1/2}$ at the critical load. Similar to adhesion index, interface toughness cannot be observed directly. It is possible to obtain this parameter by measuring the scratch width and the load and the availability of critical load.

From equation 3, if L is the critical load, d is scratch width and t is the thickness of coating, K_2 is interface toughness of the coating, it is obviously shown that Interface toughness is not a function of coating thickness.

Interface toughness for specimen observed is presented in table 4. It is clearly shown that the interface toughness for single coating is lower than that for duplex coating. Duplex nickel coating has the highest interface toughness. Interface toughness of duplex chromium nitride coating is higher than that of duplex titanium nitride coating. Interface toughness of copper coating is the lowest.

CONCLUSION

Critical load for metallic coating and ceramic coating have been calculated using three methods namely direct method, intercept method and scratch width method. In addition, adhesion index and interface toughness of coatings have also been calculated. The results show that critical loads, adhesion indexes and interface toughness of duplex coatings have higher than that of single coatings. Duplex nickel coating is the highest while copper coating is the lowest critical load, adhesion index and interface toughness

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