Military Technical College
Kobry El-Kobbah
Cairo, Egypt

10th International Conference
On Aerospace Sciences &
Aviation Technology

EFFECT OF PLASMA SPRAYED UNDER LAYER COATING THICKNESS VARIATION NI AL95-5 ON THE TOP LAYER ADHESION- AL₂O₃, TiO₂

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ABSTRACT

The effect of plasma sprayed under layer coating (Ni-Al95-5) thickness variation on both adhesion strength and tribological behavior of top layer ceramic coating (Al₂O₃ TiO₂) was studied. Series of microscopic investigation were carried out on both under layer and top layer coatings of test specimens, which were manufactured from base metal alloy steel 15Cr Ni 6, to measure the porosity, unbounded particles, interfacing and oxide particles content percentages which were found within the permissible limits of ASTM specification. Also, a series of coating tensile adhesion strength tests and wear experiments carried out using the specified specimens. The microhardness of layers structure are reported. A relation ships between the adhesion strength, microhardness of the two layers and wear losses against the underlayer thickness variation were predicted. From these curves and measurement, the optimum plasma sprayed under layer coating thickness for the top layer plasma sprayed ceramic coating was determined.

KEY WORDS: Plasma sprayed coating, Plasma sprayed underlayer coating, Plasma sprayed top layer coating, Adhesion strength, Micro hardness.

1. INTRODUCTION

Plasma -sprayed ceramic coatings, with their excellent performance in resisting high temperature, wear, corrosion and erosion have been used in many practical applications, in particular or automotive and aviation industries [1-3]. For example, spray metallic powders containing ceramic particles are deposited by plasma spraying to manufacture seal coating of hydraulic systems for automotive and jet engines.

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For achieving good adhesion properties, the availability of plasma as a means for depositing ceramic films into workpieces has encouraged the study and comparison of the variation of underlayers of Ni Al95-5. In addition to the coating technique used, the tribological behaviour of a ceramic coating also depends upon the coating material, the types of wear and the film thickness of both under layer and top layer coatings. These factors also have been studied [1, 2, 3].

In several studies, the effects of the top coating layer and thickness on both friction and wear behaviour were investigated. Bowden and Tabor [4], studied the coefficient of friction; they showed it to decrease with decreasing film thickness (top layer) until the film became very thin. Posti and Nieminen [5] studied the influence of coating thickness on life time of titanium nitride PVD coated high speed cutting tools.

Other authors related wear resistance to other properties of the deposits. For example Liu et. al [6] related wear resistance to, a part from another, adhesive strength; adhesive strength is also closely related to the microstructure of the deposits and substrate surface morphology [7]. Also through thickness cracks may develop in both under layer and top layer coating, predominantly during the loading, and delamination crack during the unloading, depending on the thickness and microstructural defect content in the coating [8, 9, 10]. The observations point to a complex interplay between the under layer coating thickness variation, adhesion strength and fracture process in a ceramic / metal system.

Accordingly, the aim of the present research is to study the effect of underlayer (Ni Al95-5) thickness variation on the adhesive strength and wear resistance of ceramic coating Al_2O_3 , Ti_2O_3 .

2. EXPERIMENTAL WORK:

2.1. Test specimen material

The test adhesion strength specimens of the machined substrate surfaces to be coated were manufactured as shown in Fig. 1 from base material 15 CrNi6 , whose chemical analysis is listed in table 1

Table 1. Chemical analysis of base material (15 Cr Ni6) alloy steel

C%	Mn%	Si%	Cr%	Ni%	S%	P%
0.14	0.5	0.25	1.6	1.5	0.03	0.03

2.2. Surface preparation (Grit blasting)

Successful application of a coating to test specimen surfaces (as shown in Figures 1 and 2) requires adequate preparation of the surface by, for example, ultrasonic cleaning, and roughening, to give the best bonding strength to the coating. A grit blasting procedure is generally adopted.

Fig. 1 Adhesion strength (bond strength) test specimen (SNECMA specification DMP14). (Two parts are required for each test.)

Fig. 2. Adhesion strength test specimen complete (according to specification ASTM C633-79)

2.3. Coating methodology

All the surfaces (as shown in figure 1) to be coated were prepared by cleaning with acetone in an ultrasonic cleaning tank for 15 min to remove any greasy material, and subsequently, grit blasted at a gauge pressure of 10 bar using an Al_2O_3 grit size of 420-710 μm . By varying the stand-off distance between the gun and the substrate, different surface finishes were achieved. The resultant roughness (R_a) was measured using a perthometer. Typical R_a data (perthometer trace) are shown in Figure 3a. The R_a data shown in figure 3b pertain to the average of four different specimens with different stand - off distances. The optimum stand-off distance for achieving the maximum R_a (4.35 μm) is seen to be in the range of 135-140 mm with spray angle 75°.

Fig. 3a. Perthometer trace of grit blasted surface

Fig. 3b. Effect of stand-off distance on surface roughness (grit blasting surface)

2.4. Plasma - Spraying process and equipment

Before plasma spraying of the ceramic, a bond coats with different thickness of Ni – Al95-5 was sprayed into the substrate surface according to the requirements and parameters mentioned in Tables 2 and 3 respectively.

Table 2. Thermal spray coating thickness of top and under layer for the adhesive strength test specimens.

Specimen No.	1	2	3	4	5	6
Specification						
Bond layer thickness (under layer thickness)	50µm	70µm	90µm	110µm	130µm	150µm
Number of specimen	6	6	6	6	6	6
Ceramic layer thickness (Top layer thickness)	100µm	100µm	100µm	100µm	100µm	100µm
Total thickness of two layers	150µm	170µm	190µm	210µm	230µm	250µm

Table 3. Plasma spraying parameters

Serial No.	Parameter	Unit	Under layer coating Ni-Al95-5	Top layer coating Al ₂ O ₃ -TiO ₂
1	Powder particle size µm	µm	45 – 90	10 – 45
2	Primary gas (Ar)	litre/min	49	38
3	Secondary gas (H ₂)	litre/min	8	9
4	Powder carrier gas (Ar)	litre/min	3.3	4.6
5	Powder flow rate	Kg/hr	2.1	2.1
6	Current	Amps	600	600
7	Voltage	Volts	55	60
8	Spray distance	Mm	140	120
9	Cooling	-	Air	Air

For deposition of the ceramic coating, a DC electric arc was used to produce an ionised gas plasma. An arc was struck a throiated tungsten cathode and a water cooled copper anode, in a gas flow argon and hydrogen. This arc was used to heat a stream of inert gas to more than 1510^3°C , leading to dissociation of the gas molecule and partial ionisation of the gas.

As the plasma exited from the gun, powder to be sprayed on to the substrate was introduced into the plasma jet, which was melted and propelled by the high velocity stream, producing a dense, well-bonded coating on the substrate.

A Bay State System (M1000) with plasma gun F4 was used for spraying and the coating parameters are shown in Tables 1 and 2. The parameters of spraying the ceramic coatings on all the substrate surfaces of the specimens which had been already sprayed with underlayer coatings Ni-Al95-5 are technically according to the parameters listed in table 1, 2.

2.5. Hardness Test:

The coatings were tested for Vickers Hardness with Vickers Hardens tester (Type-DM) with load range of 10, 30, 50, 100, 300 grams. With this instrument, the hardness is measured in the two layers (under layer and top layer) with 30 gr. The indentations of hardness tester for the two layers were photographed in order to show the hardness value variations.

2.6. Metallography investigation:

One specimen from each under layer thickness listed in Table 3 has been cut perpendicular to the two plasma spray layers. These pieces have been embeded in plastic mould so as to check the porosity, the unbonded particles, the interface, the oxidation and cracks. These investigations carried out by using a camera equipped optical microscope with magnification up to x1000.

2.7 Adhesion strength test:

Each two adhesion strength test specimen with the same under layer coating were assembled using adhesive material FM 1000 as shown in Figure 2. The geometrical conditions were achieved by using a specially designed fixture. They were then installed inside an electrical furnace which was adjusted to 170°C for duration of 2 hours. After cooling, the adhesived specimens were installed on the tensile strength machine one by one. The tests were run by using a universal tension machine with a 300 KN capacity equipped with a special adapter designed for the adhesion strength test.

2.8. Wear Test

The ceramic coated pins were subjected to a sliding wear test, in a standard pin-on-disc machine [11], over a range of sliding speeds from 0.3 to 10 m/s with normal loads from 5 to 50 N. The disc material was AISI316L stainless steel, hardened to 55 SHRC. To study the wear behavior of top layer coating ceramic with under layer variation Ni Al95-5, the weight losses of the coated pins were measured to an electronic balance with an accuracy of 0.01 mg.

All tests are considered as comparative tests which have been run under normal load 10 N and sliding speed 4 m/s, fixing an emery paper (600 grit) to the rotary disc for different durations.

2.8.1. Wear test specimen

The coated pins were manufactured from the base metal 15 Cr Ni 6 as shown in Figure 4, whose chemical analysis is listed in Table 1.

3. Results & Discussion

Table 4 shows the microstructure measurements for the specimens of the different under layer thickness listed in Table 2.

Fig. 4. Wear test specimen

The microscopy measurements of porosity (percentage and diameter), unbonded particles (percentage and average of unbonded diameter), interfacing between base metal, under layer and top layer coatings, oxide particles, cracks and hardness H_{vo.3} of the two layers are listed in Table 4 for all test specimens. The porosity percentage range in the perpendicular cross section unit area of under layer is from 5% to 7%, meanwhile the porosity diameter values are from 10 μ m to 12 μ m. Also, the percentage of unbonded particle in the unit area of under layer coating is within 5%, but the unbonded particle diameter in is from 20 μ m to 30 μ m. In addition, the interfacing percentage of the under layer coating per unit area is ranging from 10% to 20%. On the other hand, the microscopic investigation shows that both under layer coatings and top layer contain the permissible oxides values (according to the ASTM E C633-85) and are free from cracks.

Table 4. The micro structure measurements of both top layer and the under layer thickness variation specimens.

Serial No.	Layer thickness	Porosity		Unbonded particle		Interfacing	Oxide	Cracks	Hardness Hvo.3
		Percentage %	Average diameter	Percentage %	Average diameter				
1	Top layer 100µm	> 5%	10µm	Free	Free	O.K	O.K	Free	760
	Under layer 50µm	≅ 5%	12µm	<5%	20µm	15%	within limit	Free	160
2	Top layer 100µm	≅ 7%	10µm	Free	Free	O.K	O.K	Free	750
	Under layer 70µm	5%	10µm	5%	20µm	10%	within limit	Free	170
3	Top layer 100µm	5%	10µm	Free	Free	O.K	O.K	Free	740
	Under layer 90µm	5%	12µm	5%	30µm	15%	within limit	Free	160
4	Top layer 100µm	7%	10µm	Free	Free	O.K	O.K	Free	750
	Under layer 110µm	5%	12µm	<5%	30µm	15%	Within n limit	Free	170
5	Top layer 100µm	5%	10µm	Free	Free	O.K	O.K	Free	750
	Under layer 130µm	5%	12µm	<5%	30µm	15%	within limit	Free	170
6	Top layer 100µm	7%	10µm	Free	Free	O.K	O.K	Free	780
	Under layer 150µm	5%	12µm	<5%	25µm	20%	within limit	Free	170

Figure 5 reveals the vicker hardness Hr0.3 values for both top layer and under layer against the under layer thickness. However, the top layer $Al_2O_3-TiO_3$ shows the greatest hardness when compared to the under layer coating Ni-Al95-5 as mentioned in table 4 and shown in Figure 5. Also, it is noticed that the vicker's indentation traces in both top layer and under layer reveal the variation of hardness as shown in Figure 6.

Fig. 5. Vickers Hardness variation for both layers against under layer thickness.

Fig. 6. Interfacing and Hardness indentation in under layer.

The small variation in hardness for $\text{Al}_2\text{O}_3\text{-TiO}_3$ and Ni-Al95-5 is probably due to less porosity content, less unbonded particle and the less oxides.

Fig. 7. The average tensile strength against the under layer coating thickness.

Figure 7 shows the relationship between the average tensile strength of adhesion test specimens and the under layer Ni-Al95-5 thickness variation. The bond strength increases slightly with increase in the under layer thickness.

The specimen with under layer coating of 110 μm thickness gives maximum tensile strength 42 Mpa. As the thickness of the under layer coating increases, the tensile strength values decrease rapidly as shown in Figure 7.

Figure 8 shows the sliding behaviour of ceramic-steel pairs (wear loss against sliding distance) for six under layer coating thickness variation (from 50 μm to 150 μm at a load of 10 N and sliding speed of 4 m/s. Wear loss reaches a maximum in specimen with under layer thickness of 50 μm and 70 μm respectively

As shown, the specimen of under layer thickness 110 μm achieved lower wear losses and followed by these of under layer thickness 90 μm , 130 μm and 150 μm . The rate of wear loss initially increases to a sliding distance 600 m, after which steady wear loss between 600 m to 3500 m is observed. The higher wear losses in the initial stage up to 600 m may be due to removal of surface asperities.

On the other hand, it is evident that the specimens with under layer Ni-Al95-5 with thickness of 110 μm give higher friction coefficients and lower wear loss. This behaviour can be attributed to the optimum under layer thickness of 110 μm . This layer thickness of top layer (ceramic) coating shows evidence of adhesion mechanisms at work, such as plastic deformation, transfer film formation, localized burst out and grains pull out, in the sliding wear of ceramic-steel contacts.

Fig. 8. Effect of sliding distance on wear loss

CONCLUSION

- 1- Comparing bond strength of the six under layer coating thickness variation shows a maximum bond strength of 110 μm under layer thickness (Ni – Al95-5) and followed by these of 90 μm and 130 μm .
- 2- In the dry sliding wear test, conducted on a pin-on-disk machine over a load 10 N and sliding speed 4 m/sec, the ceramic coating shows the best wear resistance with under layer coating thickness of 110 μm followed by these of 90 μm , 130 μm and 150 μm .

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