Nickel Coating for Thermal Management Application

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Abstract:

Nickel coating is extensively used as protective coatings, decorative coatings and various engineering applications. Generation of suitable protective coating for thermal management of metallic structures is a demanding area in aerospace application and other advanced engineering fields.

In this study, the use of electro-deposited nickel has been investigated as a protective coating to metallic components, which experience moderate to high temperature along with thermal fatigue. For this purpose, nickel coating of lower hardness (240-270MHV) was developed with optimized parameters. Its protective value and the fatigue resistance in moderate to high temperature (up to 1100K) were evaluated by functional tests like thermal shock test and Low Cycle Fatigue (LCF) test. In addition, standard qualification tests like Heat Quench, Bend Test, Porosity, Surface finish and Micro-hardness tests have been carried out to ascertain the quality of electro-deposited Nickel. The strain evaluation on Bend test specimen was also done by FEM Analysis. Related test details and results are discussed in this paper. Nickel coating by electro-deposition is found to be a promising candidate in thermal management applications for better protection with fatigue resistance.

Keywords: Nickel plating, Low Cycle Fatigue Test, Micro-hardness, Thermal Shock, Strain evaluation

1. Introduction

Nickel plating process is extensively used for decorative, engineering and electroforming fields and its engineering applications are mainly for improvement of corrosion/wear resistance, salvage or buildup of worn out/undersized parts, modification of magnetic properties, preparation of surfaces for top coats, etc. These applications exist in the chemical, nuclear, telecommunications, aerospace, consumer electronics and computer industries (1).

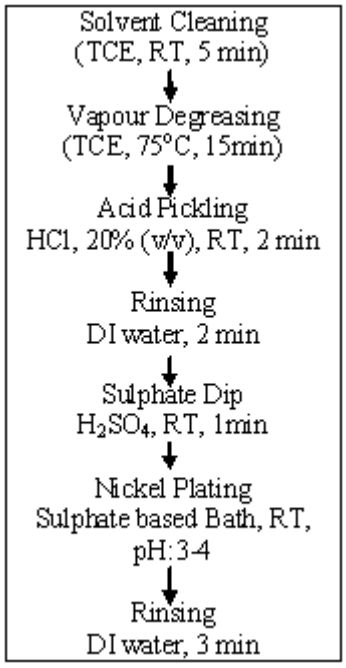
Development of suitable protective coatings on metallic parts for thermal management is a demanding requirement, especially in aerospace and aviation industries, where engine parts experience high temperature and thermal fatigue. Due to high thermal conductivity, copper and its alloys find extensive use in thermal management applications (2). In some cases in order to protect the base material from high heat flux, suitable protective coating is essential (3). This study attempts to explore the possibility of establishing a metallic coating for thermal management application. Owing to its poor thermal conductivity and high melting point, Nickel was selected as the candidate for the investigation, in this study.

The copper alloy, which is being used in high temperature applications, is selected as the substrate material. Copper alloy specimens with machined surface with Ra value ranging from 2.887 to 2.274μm were used for the study. The selected dimension of the standard specimen was 60 x 60x 5mm. The initial weight of each specimen was also measured.

Being a convenient and economic method, that can produce a pure, uniform, non-porous coating without any apparent defect, electro-deposition was selected as the coating method. Since, the purpose of the coating in this study is to protect

the base material from heat flux and thermal shock, the coating should have enough ductility with minimum internal stress as per literature. Hence, after detailed studies, a Sulphate-based bath formulation was chosen for the proposed investigation. The coating thickness was selected in the moderate range of 150-175µm for the initial phase of studies and it was decided to modify/ optimize it further, based on the test results, if found necessary. The surface finish of the coating is also critical as the thermal properties vary with respect to topography of the coating. Hence it was aimed to generate a coating of Ra value 0.025 to 1.6µm (▽▽▽)

Table-1: Process sequence



For the qualification of the electro-deposited nickel coating, tests recommended by ASTM standards were followed. In order to evaluate the functionality of the coating at high temperature/ for thermal management, two tests were identified - Low Cycle Fatigue Test (LCF) to explore the fatigue resistance of the coating at thermal loads and Thermal Shock test to evaluate the thermal resistance of the coating-substrate interface. This paper deals with the related tests carried out on Nickel plating to examine its suitability as a protective coating for thermal management application.

A. Experimental Details:

The sequence of process followed to generate a ductile nickel coating is given in Table-1. The substrate copper alloy specimens were thoroughly cleaned with Trichloroethylene solvent at ambient temperature for five minutes, followed by vapour degreasing with TCE at 75°C for 15 minutes. The cleaned specimens, after cooling, were pickled in HCl bath at RT for two minutes. After thorough rinsing with De-Ionised (D I) water, a sulphate dip was given in 10% Sulphuric acid solution for one minute. The specimens were loaded in plating bath after thorough rinsing in DI water. Bath composition and parameters were selected in the middle range of the specified values. The coated specimens were inspected for its appearance, thickness, surface finish and hardness as preliminary tests. With the middle range of composition and parameters, the hardness obtained was 300-330MHV. On the functional tests with the coated specimens, micro-cracks were noticed even with the first cycle of the LCF test, which prompted to generate a more ductile coating to meet the requirement. For the second set of trials, the cathode current density was reduced by keeping the composition and other parameters the same as that of the first set of trials. With the modified current density, the micro hardness of the coating was found to be reduced to 240 -270MHV. The LCF test conducted with the reduced hardness gave promising result. Hence, detailed qualification tests were carried out on the specimens coated with the modified process and all the functional tests were also repeated to establish the consistency.

B. Testing and Characterization

For the coating qualification, total 10 tests, including two functional tests, were identified. General tests like Visual inspection, Thickness Measurement, Micro hardness measurement, Surface Finish measurement, Adhesion Tests (Heat Quench, Bend Test, Thermal cycling) and Porosity test were followed as general qualification tests while Thermal resistance by Thermal shock test and Thermal fatigue resistance by LCF (Low Cycle Fatigue) were done to evaluate the functionality of the coating. Three coated specimens were used for each test. Out of these

ten tests, thickness, micro hardness and surface finish measurements were quantitative in nature. The test matrix is given in Table-2 and the details of each test are described below.

Table-2: Test Matrix

Characteristics	*Proposed Tests*	*Ref Document*	*Method/ Conditions*	*Acceptance Criteria*
Appearance	Visual	ASTM B689-97 ASTM B456-11	Naked eye+ 4x magnification	No surface defects
Thickness	Thickness Measurement	-	Microscopic method & Weight Difference	150-175µm
Adhesion	Heat Quench Test	ASTM B 571-97	Temp: 250°C- RT Time: 10'; Quench Media: Water	No surface defects such as crack, blisters, peel off, de-bonding etc.
	Thermal cycling	MIL883; Method 1011	-65°C to +150°C; Soaking time:10'; No. of cycles:15	
	Bend Test (Only for Ni)	ASTM B 571-97	Bending over a mandrel of dia 4 times the substrate thickness	No debonding at the interface with a sharp object
Surface Finish	Ra value measurement	ISO4288	Profilometry	<1.6 µm
Porosity	Ferro cyanide Test	ASTM B 733-04 & B765-03	Drop test	No brown spot
Hardness	Micro hardness Test	ASTM E384-11	100/300g load, Vickers	Ni: 200-300MHV Cr: 400-500 MHV
Thermal Resistance	Thermal shock	-	Temp: 827°C RT; Time: 15' Quench Media: Oil	-do-
	Low Cycle Fatigue Test		0.4% strain, 790K	No crack, peel off, blisters etc.

Visual Inspection (as per ASTM B 689-01): Visual inspection was carried out on all the specimens after Ni plating, for uniform appearance, absence of any surface defects like uncoated areas, blisters, pits, roughness, cracks, flaking, burned deposits and nodules.

Coating thickness measurement: Two methods such as Weight difference and Microscopic method were used to measure the coating thickness. By weight difference method, average coating thickness value is calculated using the equation;

$$T = [\ddot{A}W/(A \times d)] \times 10^4\ \mu m$$

where, ÄW is the weight difference of coated specimen in grams, A is the area of coated surface in cm^2 and d is the density of coating in g/cc.

In microscopic method, as per ASTM B 487, the coated specimens were cut, moulded, polished and etched. The thickness was measured using an optical microscope (Make: Olympus, Model: SC30).

Adhesion Tests: The de-lamination/peel off resistance of the coating to the thermal/mechanical shock indicates the adhesion property of the coating. The following tests were conducted to find the adhesion property:

Heat-Quench Test, as per ASTM B 571-13: 3 Nos. of plated specimens (60x60x3.5mm) were heated in an oven at 250°C for 20 minutes and quenched in water at room temperature. After completion of the heat-quench test, visual inspection and Tape test were repeated.

Bend Test, as per ASTM B 571-13: Three specimens (200 x 25 x 1mm) were coated selectively on one side, with nickel. The specimens were bent over a mandrel of 4mm dia. with the coated surface away and until its two legs became parallel. The deformed area was visually inspected with 4X magnification. Subsequently the strain generated was analyzed with 3D finite element model using multilayered PLANE181 shell element available in ANSYS.

Thermal Cycling as per MIL 883, Method 1011: Three coated specimens were loaded inside the Thermal shock chamber (Make: M/s Angelantoni Kasco Pvt. Ltd) and the test was carried out with 15 cycles, each step having 10 minutes of soaking time. The temperatures at the hot zone and cold zone are +150°C and -65°C, respectively.

After the test, the specimens were visually examined for any peel off or flaking and subsequently subjected to tape test. A 25 mm wide semi–transparent, cellulose regenerated pressure sensitive adhesive tape (3M 250) of length 75 mm was cut afresh from the roll and stuck over a smooth clean dry area selected on the coated specimen, ensuring that the tape was devoid of wrinkles and entrapped air bubbles. Removed the tape by seizing the free end and pulling it off rapidly back upon itself at an angle as close to 180° as possible. The surface was tested for any peel off/flaking of the coating.

Surface Finish Measurement: The surface finish of the coated specimens was done by evaluating the surface finish (Ra value). (Equipment: Form Talysurf, Make: Taylor Hobson, Model: PGI 800)

Porosity Test, as per ASTM B 733-15: The coated specimens were wiped with glacial acetic acid. After 3 minute, applied a solution of Potassium ferrocyanide, prepared by dissolving 1g Potassium ferrocyanide and 1.5g of methyl cellulose in 90ml of boiling distilled water. The solution was kept for 2 minutes. The tested surface was inspected for any decolouration or brown spot that indicates porosity.

Micro-hardness Measurement, as per ASTM B578-87: Micro hardness of coating was measured by Micro hardness Tester (Make: Omnitech, Model: MHV –V). Test Load: 100g, Magnification: 500X.

Thermal Shock Test : The thermal resistance of the coating was tested by this method. The furnace was preheated to 1100K. Four specimens were loaded inside the furnace for 15 minutes so that the specimen temperature reached the furnace temperature. A thermocouple was placed on one of the specimens. The specimen with thermocouple was cooled with forced air. Other three specimens were quenched in oil at RT. Subsequently visual inspection and tape tests were carried out.

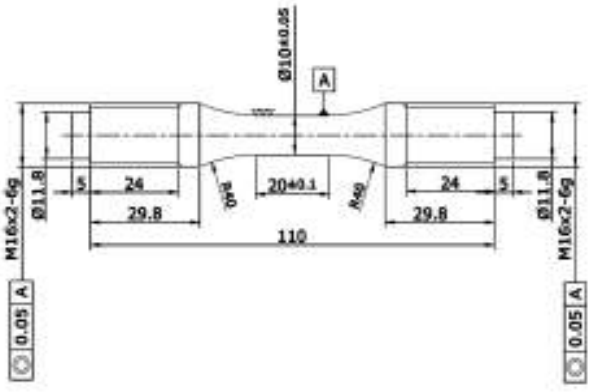


Fig. 1: LCF Specimen

Low Cycle Fatigue (LCF) Test: Fatigue resistance of the coating at high temperature was measured by LCF test (4,5). The test was carried out on a cylindrical tensile specimen with combined tensile and thermal load. The specimen dimension is given in Fig.1. Normally, the strain experienced by the substrate material in the intended application varies from 0.4 to 0.6%. Hence, the test load was selected to this range. At first, the coated specimen was tested by inducing a total strain of 0.6% at RT. The specimen was unloaded and inspected thoroughly to confirm that it is crack-free. The same crack-free specimen was subjected to a heating cycle, with the specimen heated to 760K and then cooled to ambient temperature. The specimen was then inspected thoroughly to confirm that it was crack-free. Then it was heated gradually to 760K and then subjected to tensile test, inducing a total strain of 0.4%. After each cycle, Dye Penetrate Test (DP) was done to detect the crack. The number of cycles employed was 15.

C. Results & Discussion

Visual inspection: All the coated specimens (22 Nos.) were visually inspected by naked eye and with 4X magnification and found to be free of defects like uncoated areas, blisters, pits, roughness, cracks, flaking, burned deposits and nodules.

Coating thickness measurement:

Table-3: Coating thickness of specimens

Specimen Nos	Thickness (in µm)		
	Readings	Range	Average
Specimen 1	162.56, 164.48, 168.40	162 To 168	165.15
Specimen 2	163.84, 164.48, 164.48, 166.40	164 To 166	164.98
Specimen 3	164.48, 166.40, 167.68,167.68	164 To 167	166.56

The thickness of all the specimens was measured by calculating the weight difference with Scientific balance (Make: Scientec, Model: SA 510, Accuracy: 0.1mg). The thickness range was from 156 - 167 µm against the specification of 150 ± 25 µm.

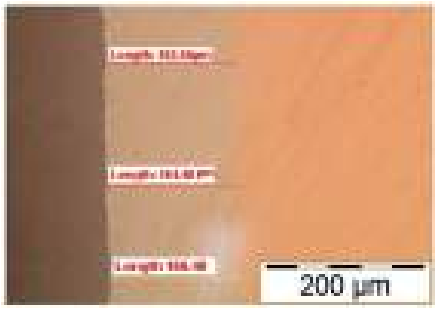


Fig.2: Cross section of coating

Three coated specimens were cut, moulded, polished and etched. The thickness was measured by microscopic method, using an optical microscope. (Make: Olympus, Model: SC 30). The thickness obtained was within the specified limit (162 to 168 µm). The cross section is given in Fig. 2 and the readings are tabulated in Table-3.

Adhesion tests: There was no crack/flaking/peel off of the coating noticed after Heat-quench and Thermal cycling test, which indicated excellent adhesion of coating to the substrate.

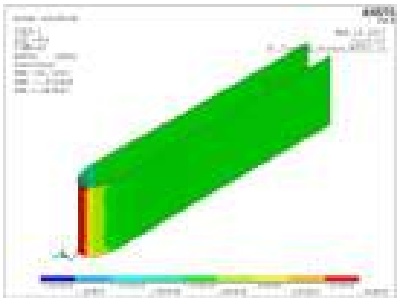


Fig.3: Meridional total strain for bend angle 180°

After bend test, hair line crack was formed on the coating due to tensile load. But the coating did not peel off /flake from the substrate while tried to lift off the coating with a sharp knife. The finite elemental analysis with multilayered PLANE181 shell element available in ANSYS showed that meridional total strain and plastic strain values were 24.45% and 24.32% respectively for the bend angle of 180°. Maximum equivalent stress value for a bend angle of 180^0 is 438.9N/mm2. Images for total strain and maximum equivalent stress for bend angles 180°

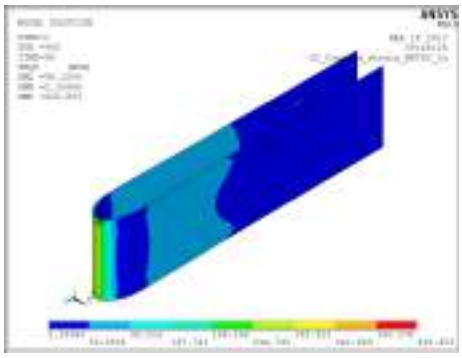


Fig.4: Maximum equivalent stress for bend angle angle of 180°

are shown in Fig.3 and Fig.4.

Surface Finish Measurement, as per IS 3073: Ra value of the coating was measured by Profilometer. (Form Talysurf, Make: Taylor Hobson, Model: PGI 800). The Ra value obtained was 0.25 to 0.74 µm, which was well within the specification of <1.6 µm

Porosity Test: There was no brown spot observed on the tested surface indicates the non-porous coating.

Table-4: Micro hardness readings

Micro-hardness Values		
Readings	Range	Average
242.5, 241.2, 257.9, 269.3, 270.7, 244.0	241-270	254.26

Micro hardness Measurement: Micro hardness of coating was measured by micro hardness Tester. (Make: Omnitech, Model: MHV –V). Test Load: 100g, Magnification: 500X. The obtained hardness values are 240 to 270MHV. The readings obtained are given in Table-4.

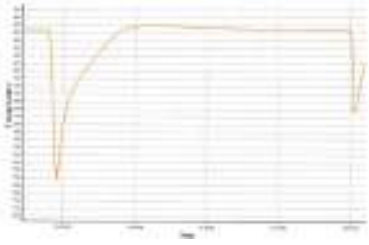


Fig.5: Temperature data of thermal shock test

Thermal shock Test: This test was conducted to evaluate the effect of thermal shock on the coating. After the test at 1100K and quenching in oil at RT, there were no blistering or flaking noticed on the coating (Equipment: Electric Box Furnace, Capacity: 36KW, Make: M/s Thermosystems). The adhesion was tested with subsequent tape test. Hence, the thermal resistance of coating up to 1100K was established. The temperature data of thermal shock test is given in Fig. 5

LCF Test: LCF test was conducted to evaluate the fatigue resistance of the coating at moderate to high temperature. The test cycle was continued until the coating started cracking. Crack was observed on the coating only after 20 cycles and that was not appreciably grown up to 30 cycles. Further, it was started to increase. The test was continued up to 40 cycles. The formation of crack and its growth was evident by the DP test at each stage.



Fig6: Images of LCF Tested specimens after DP test

Initially this test was conducted with Nickel coating having hardness value of 300-330MHV, as well and crack was found to be initiated with the first cycle of the test itself. On further cycles, it was found to be aggravating. The images of LCF tested specimens with both the hardness values after DP test is given in Fig. 6.

The functional tests results ensure that Nickel plating is a suitable protective coating for thermal management application.

D. Conclusion

1. Nickel coating with ideal hardness for thermal management application was established.
2. The various qualification tests like visual, thickness, micro- hardness, surface finish, adhesion tests (Heat Quench, Thermal cycling and Bend Test) and porosity were carried out successfully.
3. The strain evaluation of the bend tested samples showed a meridional total strain and plastic strain values were 24.45% and 24.32% respectively for the bend angle of 180°. There was no de-lamination from the substrate at this strain, which indicates very good adhesion.
4. In order to evaluate its suitability to use in moderate to high temperature service conditions for thermal management application, its fatigue resistance and thermal shock resistance was tested.
5. The LCF test results of specimens coated with two different hardness of Nickel indicate that the coating with a lower hardness value has a better crack resistance at thermal cyclic loads. Hence, the improvement in ductility of coating provides a better fatigue resistance to the coating.
6. The Nickel coating is a promising candidate as a protective coating for thermal management applications.

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