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ELECTROLESS NICKEL PLATING ON BERYLLIUM

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Electroless nickel plating on beryllium can be done using different nickel baths. It is proved that very adherent electroless nickel deposits of 100 μm or more thickness could be obtained from hypophosphite based electroless nickel bath.

INTRODUCTION

Aerospace, aircrafts and automotive industries are using many parts of light alloys with good mechanical properties to reduce the weight. Many of these are fabricated from light metal alloys of aluminium or beryllium, which are die cast. Under normal conditions Be is a corrosion resistant metal, but not under all conditions. This can be improved either by cladding Be with thin protective layers of suitable corrosion resistant metals or by electrodeposition. The chemical and electrochemical reactivity of beryllium make it unacceptable for plating from acid or alkaline baths. But the similarity of Be to Al and Mg suggested that the methods of plating used on these metals with suitable changes in preconditioning treatments can be employed with success. A number of approaches are available to a practical plater and they are zincating or tinning, anodic oxidation and direct plating of electroless metals [1-6].

Flow chart 1 schematically represents the possible variations in preparing Be surface for electroplating. The advantages of electroless nickel deposits on light metals like Al and Be for precision machined optical engineering devices make it one of the best materials for coating. It can be heat treated to high hardness values to impart excellent polishing and precision turnable characteristics. Other advantage is that it can be deposited with uniform coverage and thickness all over the surface of complicated parts like parabolloids, and hyperbolloids. The presence of phosphorous in the alloy enhances corrosion resistance of the coating to an appreciable extent.

To achieve adherent electroless nickel deposition on Be two process sequences have been reported namely, the direct plating and plating on a zincated surface with proper preconditioning steps. In this paper authors have studied in detail the direct electroless nickel plating on Be to get adherent coating tested the adhesion of the coating by thermal cycling or by buffing.

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FLOW CHART 1

PLATING ON BERYLLIUM

DIRECT PLATING	PRELIMINARY PREPARATION	ZINC IMMERSION METHOD
Anodic pickle	As machined Be	Zinc immersion
↓	↓	↓
Chemical pickle	Solvent degrease	Rinse
↓	↓	↓
Rinse	Cathodic alkaline cleaning	Copper plate
↓	↓	↓
Acid dip for plating from alkaline bath	Rinse	Out gas
↓	↓	↓
Rinse	Chemical polish	Cyanide dip
↓	↓	↓
Electroplate	Rinse	Rinse
↓	↓	↓
Rinse	Out gas	Electroplate
	↓	↓
	Chemical activate	Rinse
	↓	
	Rinse	

EXPERIMENTAL

The beryllium samples supplied by M/s Beryllium Machining Facility, Vikram Sarabhai space centre were in the form of rings and discs. The samples were degreased by trichloroethylene. Various chemical etching solutions [7-9] were tried for getting good adhesion of electroless deposits and are listed in table I. Acid pickles or acid dips reported in literature are too many. In our investigations the following acid dips are used 1) 10% by volume sulphuric acid 2) hydrofluoric acid, nitric acid solutions with nitric acid to hydrofluoric acid volume ratio 5:1 to 5:2. 3) buffered non oxidising solutions at pH 2-2.5. A single dip or two step activating dip are also tried with the above three types of acid solutions for evaluation of degree of adhesion.

For getting electroless deposits only hypophosphite based electroless nickel solutions were tried because literature survey on plating over Be indicated electroless nickel phosphorous alloy as the preferred deposit for beryllium. Secondly from cost aspects also the hypophosphite solutions are preferable. The composition of the solutions used in this study are given in table II. The sequence of operations giving adherant electroless deposits on Be found from this study are given in the flow chart 2. Be samples after pretreatment in zincating solution were nickel plated to get a thickness of

100 μm. Thickness of the deposit was found out by weight gain method. The rings and discs supplied are in regular shape. Hence area of the samples were calculated by measuring radii and height of the specimen. For thick deposits (greater than 25 μm) this method is sufficiently accurate, simple and straight forward. With a good micrometer least count thicknesses can be directly read by taking measurements before and after plating. The following tests were carried out for testing the adhesion of the deposit with the basis metal. Phosphorous content of the deposit was analysed by alkalimetric method [10].

TABLE I

ETCHING SOLUTIONS COMPOSITION

Zincating	:	Zinc sulphate	22 g/l
		Sodium hydroxide	160 g/l
		Tartaric acid	53 g/l
		Potassium fluoro zirconate	1 g/l
Chemical polishing	:	Phosphoric acid	75% by wt.
		Sulphuric acid	5% by wt.
		Chromic acid	5% by wt.
		Water	15% by wt.
		Temp.	40°C
Acid etching	:	Nitric acid	5% by vol.
		Hydrofluoric acid	1% by vol.
		Room temperature, 30 secs.	

TABLE II

ELECTROLESS NICKEL BATH COMPOSITIONS

Nickel chloride hexahydrate	35 g/l
Sodium hypophosphite monohydrate	20 g/l
Lactic acid	15 ml/l
Temperature	90°C
pH	4.5 - 5.0
Nickel sulphate hexahydrate	40 g/l
Sodium citrate	50 g/l
Sodium hypophosphite	35 g/l
Temperature	70°C
pH	7.0-7.5
Nickel sulphate hexahydrate	30 g/l
Sodium citrate	60 g/l
Sodium hypophosphite	25 g/l
Ammonia	10 ml/l
Temperature	80°C
pH	10.0

FLOW CHART 2

PLATING ON BERYLLIUM:

Beryllium rings and discs as received
Degrease in trichloroethylene
↓
Soak clean in alkaline cleaner at 35°C for 3 minutes
↓
Rinse in running water
↓
Rinse in running water
↓
Neutralising dip in 1:1 nitric acid solution at RT for 60 secs.
↓
Rinse
↓
Zincating
↓
Remove zinc deposit in 50% by volume nitric acid
↓
Rinse
↓
Zincating
↓
Dip in 50% by volume nitric acid for 30 sec, at 30°C
↓
Rinse
↓
Electroless nickel plating to desired thickness
↓
Rinse
↓
Dry

Thermal cycle test :

This test is similar to thermal cycle tests for plated plastics except that the sample is heated to 150°C in an air oven and kept at that temperature for one hour. The hot sample is then plunged into ice cold water at 0°C. This cycle is repeated thrice if no blister appeared in previous cycles. Adhesion is good if there is no blistering after three cycles.

Buffing test :

The plated sample is polished mechanically on a polishing wheel rotating at high speeds with good pressure. During buffing operation local heat is produced and cause blistering of deposit if the adhesion is poor. In this investigation the electroless nickel plated beryllium samples are polished to a very high lusture and while still hot plunged suddenly into ice cold water. The samples are inspected for blistering or flaking.

RESULTS AND DISCUSSION

Rate of metal removal is insignificant in all the other preconditioning steps except in the chemical polishing step. The rate of removal during chemical polishing is 21-25 mg/cm^2/min. for the solution investigated. Further the solution is generating noxious fumes and temperature should be controled properly. Dilute zincating solutions produces best result. First zincate dip attacks the most active areas and by doing so removes metal at these parts. Dipping time is 20-40 secs to get a deep grey colour. This etching serves to remove a good part of the one to two thousand Å of the oxide that was produced. A longer time in first dip is required to guarantee coverage. Second zincating has equallising effect and the deposit obtained is more uniform over the surface of the part. The time required for this step is 10-20 secs. Complex parts require a few seconds longer time.

Electroless nickel can be deposited from any of the bath listed in table II. The choice is dictated by the phosphorous content required, rate of deposition, corrosion resistance and internal stresses tolerated for the deposit in its end use. For our study bath 3 was taken and nickel plating was carried out to get a thickness of 100μm. The hardness obtained for the electroless nickel deposits are in the range of 550-620 VPN. Phosphorous content in the deposit is 6% by weight. Adhesion tests indicated that the adherent of the coating to the Be is excellant. No peeling or flaking of the deposit was observed during buffing and thermal cycling tests.

Applications of electroless nickel deposited beryllium :

Beryllium parts coated with electroless nickel and precision turned to an accuracy between 0.05 and 1.0 μm smoothness are used in parabolloid mirrors in space vehicles. Beryllium hemispheres coated with diamond turnable electroless nickel are utilised in gyroscope and they provide superior performances in space applications where high energy forces are present and low enertia are required. Highly complicated and expensive equipments in nuclear missile and space applications require some important components to be fabricated from light metals with desired set of physical and mechanical properties to suit their needs. Electroless nickel appears to satisfy most of these requirements either as a single layer or as a composit layer of two metals. Beryllium is light weight metal meeting the requirements of high temperature corrosion resistance, high melting point and high strength to volume ratio. Therefore, electroless nickel coated beryllium may find increasing applications in these fields.

CONCLUSION

The research investigation on electroless nickel deposition on beryllium rings and discs has conclusively proved that very adherent electroless nickel can be deposited to a thickness of 100 μm or more from hypophosphite based electroless nickel bath.

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