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Electroless Nickel (EN) Coating on Aluminium Cartridge Case for Releasing Bomb Application

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The motivation for this research work is to apply innovative process of Electroless Nickel (EN) coating on aluminium (Al) cartridge cases used for releasing external stores on fighter aircraft. Aluminium, chosen for its lightweight properties, electrical conductivity, non-corrosiveness, and compatibility with energetic materials, proves advantageous in defence applications due to its high strength-to-weight ratio and softer composition compared to other metals. Nickel coating is applied to enhance corrosion resistance, increase wear resistance, eliminate the cracks, blow holes and improve the overall aesthetic appeal of the cartridge. Unlike traditional electroplating methods, novel EN methodology employed here operates through an auto-catalytic reaction, eliminating the need for electricity during metal ion deposition. Before the introduction of the EN coating, the use of bare aluminium cases led to defects such as blow holes or cracks on the cartridge surface. The EN coating applied to the power cartridge case, serve as a thermal barrier without compromising the electrical conductivity of the base material. The paper discusses the successful prevention of defects through a series of trials and emphasizes the crucial role of EN coating on aluminium cartridge cases in emergency situations for releasing bombs from military aircraft. Positive feedback received from users regarding the utilization of EN coating on Al cartridge cases proves the effectiveness and reliability of the process. This process enhances the manufacturing technology to meet the specific and critical requirements of users i.e. Indian Air Force (IAF).

Keywords: Blow holes, Closed vessel, Defects, Electron diffraction, Micro-structure analysis

Introduction

Aluminium (Al) material is extensively being used in various industries and defence applications. During the development activities, Al material is used in the power cartridges for releasing the bomb from the parent aircraft. After the conduct of extensive trials, cracks and blow holes on Al case are observed. To avoid the re-occurrence of such defects, first time Electroless Nickel (EN) plating is introduced in history. It is due to high flame temperature and pressure generated by the burning of the propellant. After EN coating, cartridges are undergone a series of experimental trials. At the end of experiments, no cracks and blow holes are observed. All the experiments are carried out with the available infrastructure in the laboratory.

This research focuses on the EN process, a technique involving the deposition of a nickel alloy on various surfaces, whether metallic or non-metallic, through electro-chemical reactions. The resulting coating can range from bright to semi-bright and dull, and in this study, it is specifically applied to Al cartridge cases for defines applications, facilitating the external release of stores such as bombs from parent aircraft. The presented results stem from multiple coatings, each with a thickness ranging from 6 to 12 microns. This EN coating process proves to be both economically viable and effective in improving surface properties. It enhances corrosion resistance, thanks to its lubricity, wear and corrosion resistance, and ability to maintain an excellent uniform thickness even on intricate surface areas.[1,2] Rajguru's study delves into the investigation of EN coating on a Per Factory TM rapid prototype model built with Per Factory TM R05 material.[3] Widely recognized for its effectiveness, EN serves as a valuable method to augment corrosion and wear protection for structural materials like steel and aluminium. Its applications span various industrial domains, including automotive, electrical, chemical, pipelines, and aerospace components.[4] The EN coating is an auto-catalytic reaction process that deposits a nickel layer on either plastic components or specific metals. Its versatility extends to coating printed circuit boards to prevent corrosion of soldered joints. The EN plating technique relies on a chemical reduction

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method, leveraging the catalytic reduction of nickel ions in an aqueous solution. Unlike traditional methods, EN plating accomplishes the deposition of nickel metal on surfaces without the need for electrical energy. To build a substantial coating using chemical means without consuming the substrate, a sustainable oxidation reaction is crucial. This oxidation reaction must occur initially and exclusively on the substrate, subsequently depositing on the initial deposit.[5] The redox potential for this chemical process generally exceeds that of a metal deposited by the immersion technique. The chemical deposition of nickel metal by hypophosphite adheres to both the oxidation and redox criteria without altering the substrate's mass. The two reactions can be expressed as follows.

Oxidation: $H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + 2H^+ + 2e$, anodic reaction
Reduction: $Ni_2 + 2e$, cathodic reaction $\rightarrow NiO$

Overall reaction: $Ni_2 + H_2PO_2^- + H_2O \rightarrow NiO + H_2PO_3^-$

EN plating is applied to both non-ferrous as well as ferrous surfaces of a several shapes and sizes with regular coating that includes screw threads. Never-the-less, this type of process is more expensive than electroplating.

The EN plating method presents several advantages in contrast to conventional electroplating techniques. Unlike electroplating, the EN process operates independently of a power supply and flux density. This characteristic lends flexibility to its application, allowing for the achievement of smooth deposits irrespective of the surface hardness of the material work-piece and its geometry. EN plating is distinguished by its highly specific plating volume and thickness capacities, offering precise control over the coating thickness. This level of control proves beneficial in applications where exact coating thicknesses are critical. Furthermore, EN plating affords the option of diverse finishes, including matte, bright, and semi-bright, providing a range of aesthetic choices and functional properties tailored to specific application requirements. A notable advantage of the EN process is its ability to eliminate the necessity for complex filtration requirements. This simplifies the plating process, reducing overall system complexity and enhancing operational efficiency. In summary, the EN plating method serves as a more convenient and versatile alternative, delivering smoother deposits, precise thickness control, a variety of finishes, and a streamlined operational setup when compared to traditional electroplating approaches.

Description of the Cartridge

The propellant is extensively used in the cartridge as the main energy source for carrying out various mechanical works.[6] This cartridge is characterised by their use of propellant burning, producing combustion products with a high temperature and pressures, thereby experiencing the strains in the propellant actuated devices / structures. The cartridge used in releasing the bomb from an aircraft is known as Ejector Release Unit (ERU). It consists of a cylindrical Al alloy, the propellant charge and an ignition element (electrode, bridgewire, and ignition charge) and a closure disc. By receiving an electrical stimulus from the aircraft power supply, it gets initiated and generates the pressure, which pushes the piston of the ERU and unlocks the externally carried store. The cartridge showing the various components and photo of the cartridge are shown in Fig. 1 (a) and (b), respectively.

Background Information

Brenner and Riddel are credited with the discovery of EN coating in 1946.[(7)] Canan explored the study of EN plating on Acrylonitrile Butadiene Styrene (ABS) in Room Temperature Ionic Liquids (RTIL).[8] Igor reported a study on coating Spheroidal Graphite (SG) cast iron with chemical Ni-P.[9] Mimani investigated the electrochemical behaviour of sodium hypophosphite in EN solution bath.[10] Ibrahim used the Taguchi method in the preliminary formulation of the EN bath.[11] Ilsoon demonstrated selective EN deposition on 3-D functional and patterned surfaces at the micron scale with submicron resolution.[12] Hamid and Elkhair reported the application of Ni-P coating on aluminium for wear resistance.[13] The study showed that reinforced particles, along with heat treatment, provided satisfactory wear resistance and hardness of the deposits. Lonyuk *et al*. evaluated the effect of EN deposition on the fatigue life of aluminium alloy, revealing a significant improvement in fatigue life and corrosion fatigue performance after deposition.[14] Manna discussed plating time for EN coating on

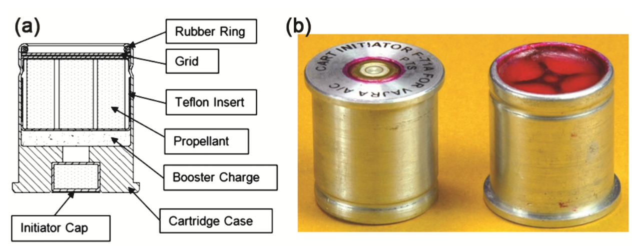

Fig. 1 —The cartridge: (a) diagrammatic image showing various components, and (b) photographic image

rubber surfaces using a citrate bath.[15] Ansari and Thakur investigated the effect of pH bath on phosphorus content in EN-phosphorous deposits.[16] They concluded that varying the pH of the EN bath could greatly improve the corrosion resistance of the coating by influencing the phosphorous content. Sahoo and Das provided a review of the tribology of EN coating.[17] The electrochemical techniques were employed by another study to investigate the effect of coating thickness and roughness on the corrosion resistance of EN coating on mild steel.[18] It was found that the coating exhibited greater electrochemical activity compared to pure nickel.

Material Testing and Properties

The choice of Aluminium (Al) as the material for the cartridge case is based on the necessity for electrical conductivity to enable the passage of current. The substrate utilized in this investigation is an Al alloy supplied as an extruded bar with a 30 mm diameter. The Al cartridge case comprises elements such as Cu, Si, and impurities like Fe, Mg, Mn, and Sn. The mechanical properties of the Al material encompass a proof stress of 300 MPa, an elastic modulus of 68 GPa, a hardness range of 123 to 139 HV, a tensile ultimate strength of 495 MPa, Poisson's ratio of 0.33, and a percentage elongation of 7.

To assess the mechanical properties, a tensile test is carried out on a dumbbell-shaped Al specimen. A stress-strain curve is generated using a Universal Testing Machine (UTM) by subjecting the test specimen to gradual loading until fracture occurs. A standard dumbbell-shaped test specimen along with engineering drawings, images of Al test specimen before and after tensile testing is illustrated in Fig. 2 (a), (b) and (c). The stress-strain graph of Al rod in tensile testing is presented in Fig. 3. The aluminium cartridge material utilized in this application demonstrates yield strength of 400 MPa.[19] These properties are acquired through testing the specimen on a UTM with the available infrastructure in the laboratory. The mechanical properties of Al test specimen are summarized in Table 1 and the elemental composition of Al material in percentages is presented in Table 2.

Experimental Procedure and Details

EN Process

In recent years, EN deposition technology has become increasingly popular in industrial applications. This process involves catalytic reduction, where nickel metal ions are reduced in hot, mildly acidic aqueous

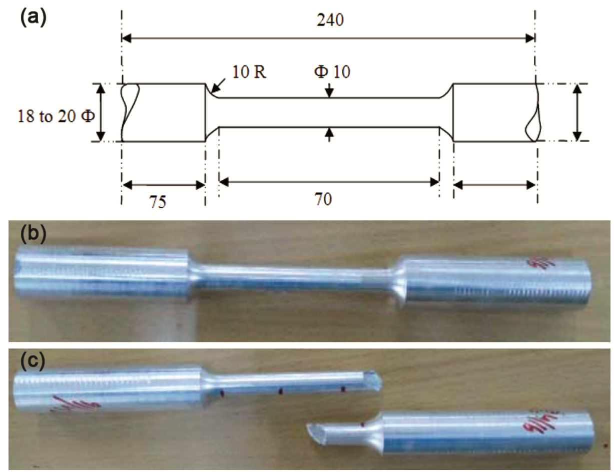

Fig. 2 — Al test specimen: (a) engineering drawing, (b) before tensile test, and (c) after tensile test

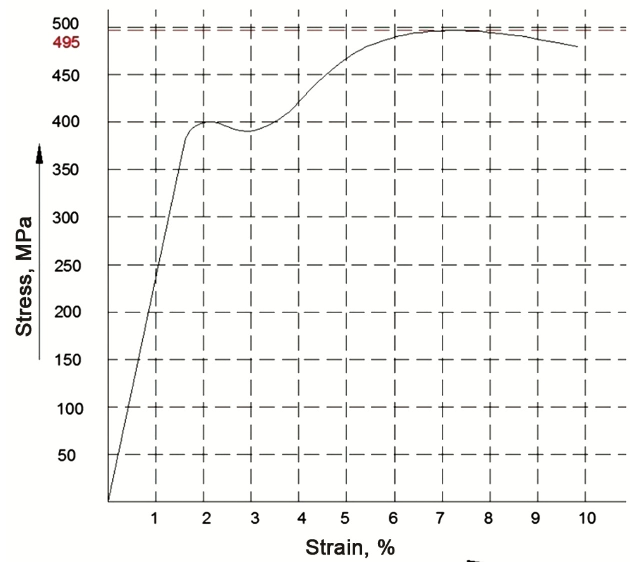

Fig. 3 — Stress - Strain curve of Al rod in tensile testing

Table 1 — Mechanical properties of Al test specimen

Modulus of Elasticity	68 GPa
Ultimate Strength	495 MPa
Yield Strength	400 MPa
Elongation	7%
Poisson's Ratio	0.33

Table 2 — Elements present in Al material

Copper	4.31%
Silicon	0.84%
Iron	0.18%
Magnesium	0.36%
Manganese	0.67%
Aluminum	Remainder

solutions at atmospheric pressure, utilizing sodium hypophosphite as the reducing agent in the EN plating bath solution. Sodium hypophosphite serves as an effective electrolyte, eliminating the need for additional supporting electrolytes. The deposition occurs in a proprietary EN solution with a specified phosphorous content, conducted at a temperature of 88 ± 2°C and a pH of 5 ± 1. Before the plating process, the material's surface undergoes a thorough cleaning using a chemical, such as sodium hydroxide, followed by extensive water cleaning. Adequate surface preparation is crucial to prevent any remaining solid particles from negatively impacting the plating quality. For Al alloys, which are exposed to air and covered by a defence oxide coating, this oxide layer must be removed before coating.[20] After applying each pre-treatment chemical, the surface is rinsed multiple times with water to ensure the complete removal of these chemicals. The de-greasing process is employed to remove oils, while scale removal is achieved through acid cleaning. Unlike processes involving electrodes or external electrical charges, the EN process is entirely chemical-based. It entails immersing the test specimen in a plating solution bath, where a reducing agent, such as hydrated sodium hypophosphite ($NaPO_2H_2 \cdot H_2O$), reacts with the material's ions to deposit the nickel alloy. The metallurgical properties of the alloy depend on the phosphorus percentage, ranging from 2–5% (low phosphorus) to 11–14% (high phosphorus). Following immersion in the Al bond and water cleaning, the test specimen is placed in an aqueous solution with a specific nitric acid ratio, maintaining a temperature of 88 ± 2°C for one hour.

The solution is continuously agitated for two hours to get the desired coating thickness of 10–12 microns. Prior to coating, all specimens underwent a zincating pre-treatment.[21] The detailed experimental procedure and steps involved in the EN process are shown in Fig. 4. The distinction between electroless deposition and immersion by comparing deposit thickness *vs.* time is illustrated in Fig. 5. Composition and conditions of electroless nickel plating on Al material is given in Table 3.

Result and Discussions

Hardness Measurement

The application of the EN coating process to Aluminium (Al) cartridge cases follows the outlined procedure in paragraph 3.1. Subsequent to experimental trials, a visual inspection of the fired cases is conducted.

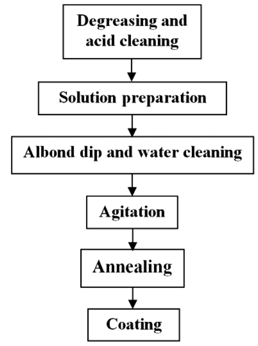

Fig. 4 — Flow diagram of EN process

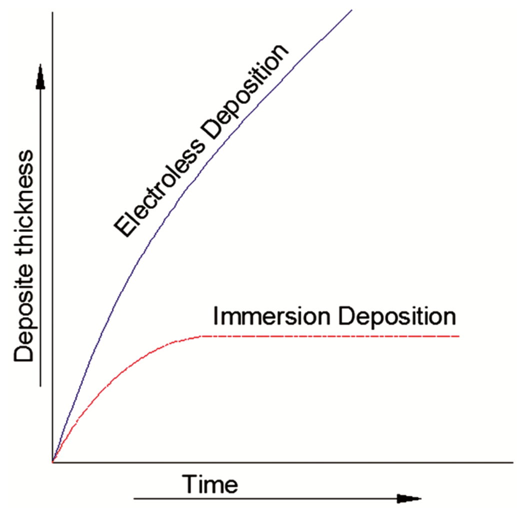

Fig. 5 — Comparison between electroless and immersion method by deposit thickness *vs.* time

Table 3 — Composition and conditions of EN plating on Al material

Chemical	Formula	Composition (g/L)
Nickel chloride	$NiCl_2$	20
Sodium hypophosphite	$NaH_2PO_2 \cdot H_2O$	15
Sodium citrate	$C_6H_5Na_3O_7 \cdot 2H_2O$	35
Ammonium chloride	NH_4Cl	30

Hardness measurements are taken at the base, middle, and tip of the cartridge case, both before and after the EN coating, as well as for fired cases. Images of the cut section of Al cartridge case are displays in Fig. 6. The entire length of the fired cases is subjected to hardness measurements.

Upon thorough analysis, it is noted that there are slight variations in the hardness from the base to the tip for cartridge cases after coating, in comparison to cartridges before coating. The hardness of the Al case post-coating experiences a reduction, attributed to the annealing process that softens the base material. Comprehensive hardness results are presented in Table 4.

Table 4 — Hardness in HV at 5 kg load for Al cartridge case

Location	Base	Middle	Tip
Al case without coating	114–121	113–115	111–115
Al case after coating	41–43	41–42	38–39
Fired case after coating	49–55	58–60	47–48

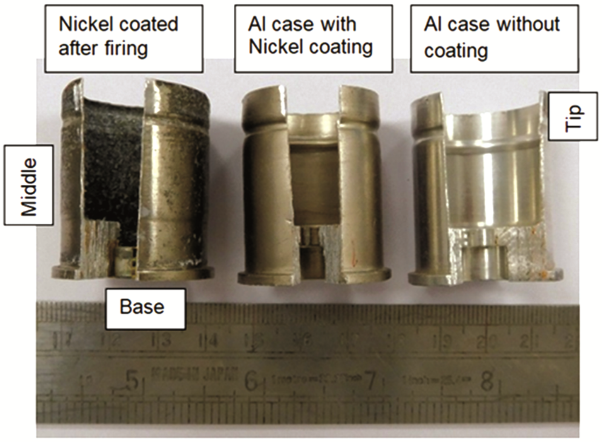

Fig. 6 — Cut section of Al cartridge case

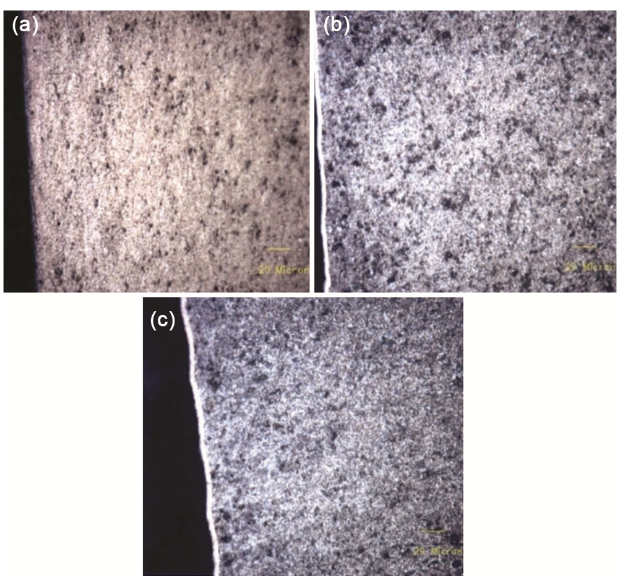

Fig. 7 — Microstructure of Al cartridge case: (a) Black particles of Mg2Si before coating, (b) Black particles of Mg2Si and nickel coating layer, and (c) Black particles of Mg_2Si and nickel coating layer after firing

Microstructure Analysis

Microstructure analysis is conducted on all three Aluminium (Al) cartridge cases. To facilitate microstructure examination, the cartridge cases are etched with HF solution and then polished. The resulting microstructure images are depicted in Figs. 7(a), (b), and (c) for before coating, after coating, and fired cases after coating, respectively. The microstructure reveals black particles of Mg_2Si under a magnification of 200X. Upon comparing the microstructures before and after firing, it is evident that there are no noticeable changes in the microstructure of the Al material.

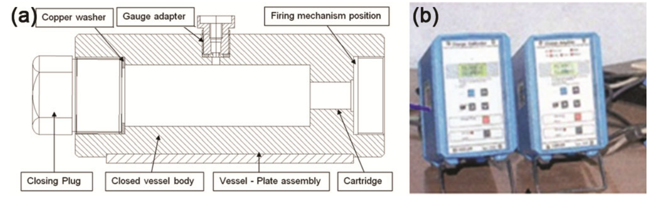

Fig. 8 — (a) Engineering sketch showing various parts of CV, and (b) Photo of charge amplifier

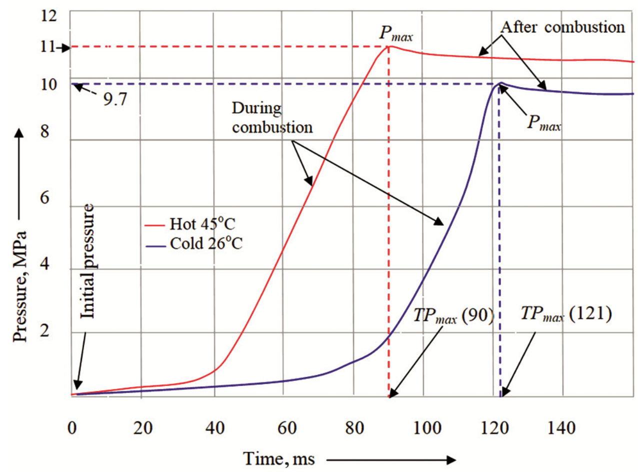

Fig. 9 — Output from CV firing

Experimental Investigation

The experimental setup for the Closed Vessel (CV) firing of the cartridge is presented in Fig. 8, comprising essential components such as a charge amplifier, scope corder, gauge adapter, closing plug, closed vessel body, copper washer, and the end plug. The engineering sketch showing various parts of CV and photo of charge amplifier is illustrated in Fig. 8 (a) and (b) respectively. To conduct the experiments, the firing mechanism is positioned at one end, while the end plug is affixed at the other end. A gauge adapter, equipped with a pressure sensor, is threaded into CV body. The cartridge is placed inside the cartridge holder. The cartridge is an electrically initiated, and the scope corder records performance parameters such as Pmax and TPmax, displayed in red for hot conditions at 60°C and in blue for cold conditions at −40°C.

The output from oscilloscope is shown in Fig. 9. Throughout the charge assessment trials, the occurrence of cracks and blow holes on the Aluminium (Al) cartridge case is noted. In order to mitigate these defects,

Fig. 10 — Images of used cartridge case: (a) without EN coating, (b) with EN coating

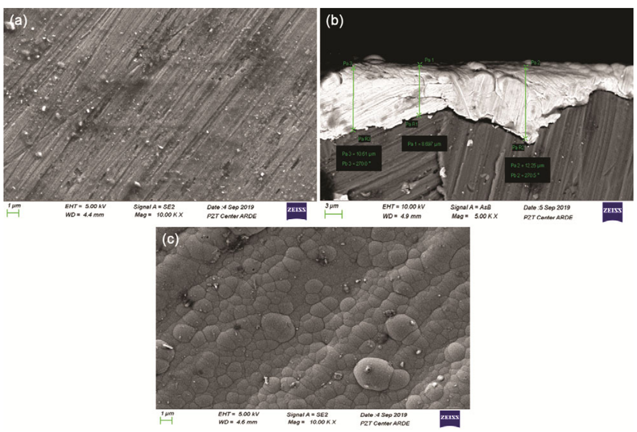

Fig 11 — SEM image of: (a) Virgin Al (grey colour) without EN coating, (b) Al with Ni-P coating thickness, and (c) surface morphology of Al

EN coating is applied on the Al cartridge case. Subsequent to the successful application of the coating on Al cartridge cases, these cartridges undergo CV firings at hot and cold temperature. Importantly, no cracks or blow holes have been observed on the Al cartridge cases following these firings. Cracks and blow holes on Al cartridge case without EN coating and visual representations of the cartridges with EN coating after undergoing a series of firings is shown in Figs. 10 (a) and (b) respectively.

Coating Thickness

The coating thicknesses on Al cartridge are measured using optical microscope and observed in the range of 9 to 10 microns.

Scanning Electron Microscope (SEM) Study

Virgin Al (grey colour) sample without EN coating with magnification 10X, Al with Ni-P coating thickness and surface morphology are shown in Figs 11 (a), (b) and (c) respectively. The apparent width of the coating is shown in Fig. 11 (b), clearly revealed that the thickness of Ni-P ranging from 8–13 µm. This is solely revealed by the photograph with a contrast between the two materials boundaries. Ni-P coating on Al was confirmed by surface morphology carried out by scanning electron microscope (SEM) as shown in Fig. 11 (b). An image of the cross-section, as revealed in a conventional electron diffraction (EDX) image, was measured. The boundaries of the coatings are seen in the cross-section image as shown in Fig. 11 (c) with surface morphology. Further, the coating thickness was confirmed by EDX image that clearly distinguishes between Ni and P elemental peaks. P (blue colour) on Al base material (olive green colour) and EDX colour image is shown in Figs. 12 (a) and (b) that defines the coating of Ni (magenta colour). EDX image with Ni-P coating is shown in Fig. 12 (a). The color mapping image of Ni (magenta colour) is depicted in Fig. 12 (b) clearly indicates the coating of Ni-P as compare to EDX image. This is confirmed by sharp peaks of Al, Ni and P (left) in the elemental analysis mode of EDX.

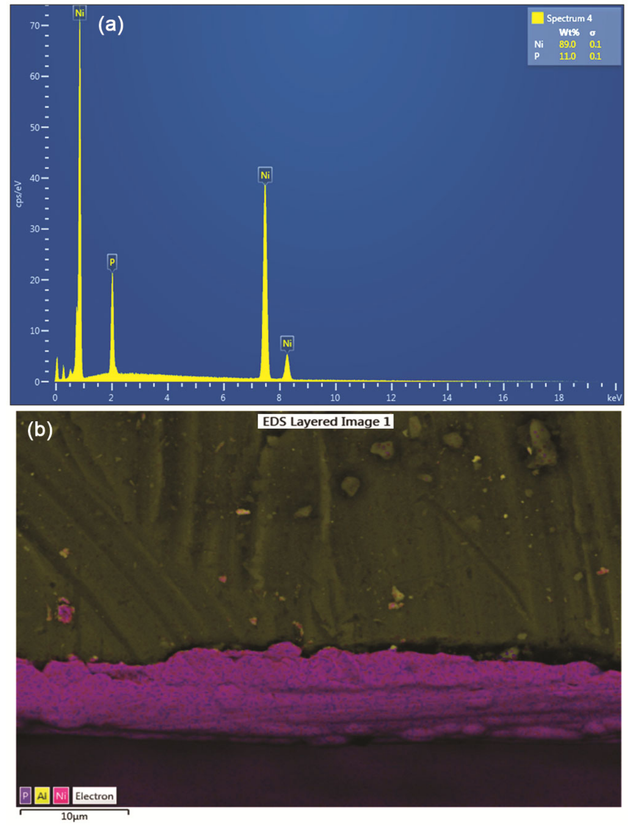

Fig. 12 — (a) Electron diffraction image (EDX) with Ni-P coating (b) Color mapping image of Ni (magenta colour)

According to this, the EN deposit thickness likely varies from 8.697 to 12.25 microns.

Conclusions

The utilization of EN plating has proven successful in coating a uniform film (less than 10 microns) on Al cartridge cases and prevents the re-occurrence of failures such as cracks and blow holes. EN coating technique has potential application in the field of power cartridge where conductivity, protection against the environment and compatibility of explosives are highly required. Following comprehensive experiments, a substantial number of cartridges have been supplied to users, meeting their critical requirements thereby keeping large fleet of aircraft in operational readiness. The detailed experimental investigation affirms the safety and environmentally friendly nature of EN plating on Al cartridge case surfaces. The EN coating eliminates the need for expensive instruments, contributing to a significant reduction in both environmental pollution and process costs. The research indicates that EN deposition holds substantial potential for various coating applications, acting as a viable alternative to conventional electroplating methods. These findings can be useful for industries looking to conduct further research on metal or non-metallic surface.

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Statement of conflict

The authors declare no conflict of interest to publish this research work.

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References

1 Zhao G L, Zou Y, Hao Y L & Zou Z D, Corrosion resistance of electroless Ni-P/Cu/Ni-P multilayer coating, *Arch Metall Mater*, **60(2)** (2015) 1003–1008, http://doi : 0.1515/amm-2015-0250.

2 Aerospace Material Specification, SAE-AMS-2404, Issue Sep 1957, Rev Jun 2003, superseding AMS 2404 D, https://www.scribd.com/document/432979487/AMS-2404

3 Rajaguru J C, Au C & Duke M, Study of electroless nickel plating on PerFactory^{TM} rapid prototype model, *J Achiev Mater Manuf Eng*, **55(2)** (2012) 782–789.

4 Rolf Weil and Konrad Parker, The Properties of Electroless Nickel, Chapter 4, https://advancedplatingtech.com/wp-content/uploads/2013/10/The-Properties-of-Electroless-Nickel-Plating-R-Weil-K-Parker.pdf

5 Glenn O Mallory – The fundamental aspects of electroless nickel plating.

6 Parate B A, Chandel S & Shekhar H, An experimental and numerical approach - Characterisation of power cartridge for water-jet application, *Def Technol*, **14(6)** (2018) 683–690. http://doi.org/10.1016/j.dt.2018.04.003.

7 A Brenner & G Riddel, Temperature coefficients for proving rings, *J Res Nat Bun Stds*, **37** (1946) 31.

8 Canan U R A Z, Effect of room temperature ionic liquids for electroless nickel plating on acrylonitrile butadiene styrene plastic, *J Mater Sci*, **25(3)** (2019) 276–280, https://doi.org/ 10.5755/j01.ms.25.3.20116.

9 Igor F, Grégory B & Yves W, Study of electroless nickel coatings on EN-GJS-500-7 spheroidal graphite cast iron, *Coatings*, **8(239)** (2018) 1–11, http://doi:10.3390/coatings 8070239.

10 Mimani T & Mayanna S M, Study of the role of sodium hyohophosphite in electroless nickel bath solution, *Proc Indian Acad Sci*, **109(3)** (1997) 203–209.

11 Ibrahim M M N, Sia C W & Ahmad Z A, Preliminary steps in formulating the optimum electroless nikel bath using Taguchi method, *J Teknol* , **37(c)** (2002) 67–74, https://doi.org/ 10.11113/jt.v37.533.

12 Ilsoon L, Paula T, Hammond & Michael F R, Selective electroless nickel plating of particle arrays on polyelectrolyte multilayers, *Chem Mater*, **15** (2003) 4583–4589, https://doi.org/10.1021/cm034554n.

13 Hamid Z, Abdel & Elkhair M T Abou, Development of electroless nickel–phosphorous composite deposits for wear resistance of 6061 aluminum alloy, *Mater Lett*, **57** (2002) 720–726, https://doi.org/10.1016/S0167-577X(02)00860-1.

14 Lonyuk B, Apachitei I & Duszczyk, Effect of high-phosphorus electroless nickel coating on fatigue life, *Scr Mater*, **57** (2007) 783–786, https://doi.org/ 10.1016/ j.scriptamat. 2007.05.015.

15 Manna M, Bandyopadhyay N & Bhattacharjee D, Effect of plating time for electroless nickel coating on rebar surface: An option for application in concrete structure, *Surf Coat Technol*, **202** (2008) 3227–3232, https://doi.org/10.1016/ j.surfcoat.2007.11.039.

16 Ansari M I & Thakur D G, Effect of pH Bath on phosphorus content in electroless nickel-phosphorus deposit, *Int J Tech Res Engg*, **2(7)** (2015) 1120–1124.

17 Sahoo P & Das S K, Tribology of electroless nickel coatings – A review, *Mater Des*, **32** (2011) 1760–1775, https://doi.org/10.1016/j.matdes.2010.11.013

18 Tomlinson W J & Carroll M W, Substrate roughness, deposit thickness and the corrosion of electroless nickel coatings, *J Mat Sci*, **25** (1990) 4972–4976, https:// doi.org/10.1007/ BF00580115.

19 Bureau of Indian Standard, *Indian Standard 733: Specification for wrought Aluminum and Aluminum alloy bars, rods and sections (For general engineering purposes)*, Third revisio (New Delhi) 1997.

20 Hajdu Juan, *Surface Preparation for Electroless Nickel Plating*, Ch-7.

21 Bureau of Indian Standard, *Indian Standard 13677 : Electroless Nickel phosphorus coating – specification*, (New Delhi) 1997.