PRODUCTION OF NICKEL CONTAINING PETG AND PLA POLYMERS FOR ELECTROMAGNETIC INTERFERENCE SHIELDING

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https://doi.org/10.37904/metal.2026.5254

Abstract

Electromagnetic interference (EMI) threatens modern electronics, driving the need for lightweight, robust alternatives to conventional metal shields. In this study, 3D-printed PLA and PETG polymers were metallized via electroless plating to provide the polymers with EMI shielding capability. Following chemical etching and palladium activation for optimal adhesion, PETG and PLA was coated with nickel (60 °C, 30 min) to form continuous conductive networks. Achieving an SE of up to ~60 dB, performance was strongly influenced by coating conductivity and continuity. Ultimately, these electroless-metallized 3D-printed polymers provide highly effective, lightweight, and adaptable EMI shielding solutions for advanced engineering applications.

Keywords: Electroless coating, electromagnetic interference shielding, nickel coating,

1. INTRODUCTION

In recent years, alongside rapid technological advancements, the high-frequency interaction between telecommunication systems and electronic devices has caused the proliferation of electromagnetic radiation to become a significant environmental concern. To mitigate the hazards posed by electromagnetic interference (EMI), there has been a growing interest in electromagnetic shielding materials. Traditional metal-based materials have been widely utilized for EMI shielding applications due to their superior electrical conductivity. However, the widespread application of these metallic materials is often restricted by inherent disadvantages, including high density, susceptibility to corrosion, high production costs, and processing difficulties. As a viable alternative, polymer-based materials have emerged as promising candidates to replace traditional metals, primarily owing to advantageous properties such as low density and cost-effectiveness [1]. Integrating polymers into electromagnetic shielding systems offers a viable alternative. 3D printing technology enables the customized fabrication of these materials for specific application scenarios. Among them, PLA and PETG are highly suitable for 3D printing due to their excellent processability. PLA is an economical, renewable thermoplastic exhibiting favorable mechanical, antibacterial, and flame-retardant properties. Similarly, PETG offers flexibility combined with robust mechanical strength and easy processability [2],[3],[4]. Despite the ease of fabricating these materials via 3D printing, the inherently poor electrical conductivity of thermoplastic polymers such as PLA and PETG severely restricts their efficacy in electromagnetic interference (EMI) shielding applications. To circumvent this limitation and impart EMI shielding properties to these polymeric substrates, surface metallization is of paramount importance. Among various metallization techniques,

electroless plating offers distinct advantages due to its facile implementation, mild processing conditions, and capability to deposit a continuous, homogeneous coating on non-conductive materials [5],[6].

The most common metallization methods are chemical vapor deposition (CVD), physical vapor deposition (PVD), electrodeposition (EPD), and electroless plating (ELP). CVD relies on the chemical reactions of volatile precursors at the gas-solid boundary to deposit a thin layer on a substrate. Depending on the operational conditions, it encompasses various subclasses, such as UHVCVD, PCVD, and MOCVD, the selection of which is primarily dictated by the specific characteristics of the substrate. [7]. Conceptually similar to CVD, physical vapor deposition (PVD) involves vaporizing a solid or liquid precursor. The resulting vapor is then deposited onto a substrate under vacuum, low-pressure, or plasma conditions, forming a thin film ranging from nanometers to micrometers in thickness [8]. Electrodeposition is a metallization technique that uses an external electrical current to reduce metal cations onto a conductive substrate [9]. Electroless plating (ELP) is a process driven by reduction reactions, based on the principle of depositing metal at a solid-liquid interface without the application of external electrical energy. In this method, metallic salt complexes are reduced directly onto the surface of the substrate material. Building upon these principles, the main objective of this study is to develop lightweight and cost-effective electromagnetic shielding materials by metallizing 3D-printed PLA and PETG substrates. Through a controlled electroless nickel plating process, this research aims to impart high electrical conductivity to these inherently insulating polymers, thereby comprehensively investigating their electromagnetic interference (EMI) shielding effectiveness.

2. EXPERIMENTAL DETAILS

2.1 Materials Processing

3D-printed PLA and PETG substrates were metallized utilizing an electroless nickel plating technique. Prior to the deposition, the samples were subjected to a two-step surface activation procedure, consisting of sensitization in a tin(II) chloride (SnCl2) solution followed by activation in a palladium chloride (PdCl2) solution. Subsequent to the pre-treatment, the substrates were immersed in an electroless plating bath with a total volume of 200 ml using distilled water. The coating process was carried out at a constant temperature of 60 °C for a duration of 30 min. The specific chemical composition of the plating bath is given in **Table 1.**

Table 1 Chemical composition of the electroless nickel plating bath

Chemical Component	Formula	Concentration
Nickel Chloride	$NiCl_2$	20 g/l
Sodium Cirate	$C_6H_5Na_3O_7$-$2H_2O$	40 g/l
Sodium Hypophosphite	NH_2PO_2	40 g/l
Ammonia	$NH_3.H_2O$	120 ml/l

2.2 Electroless Nickel Plating Process

The pre-treatment for electroless plating fundamentally consists of two steps: an initial sensitization process using tin(II) chloride (SnCl2) to facilitate the subsequent adsorption of palladium, which will initiate the deposition, followed by the actual surface activation step utilizing palladium (Pd).

$$SnCl2 + PdCl2 = SnCl4 + Pd \quad (1)$$

Maintaining bath temperature and adding a pH buffer are crucial for a stable deposition rate. ELP is a cost-effective, power-free method that ensures uniform coatings on complex, non-conductive substrates.

Furthermore, simple pre-treatments enable the co-deposition of metals (e.g., Pd and Ag), which significantly lowers the required alloying temperatures during subsequent annealing [10,11]

While electroless plating can utilize various metals, electroless nickel is widely preferred to impart electrical conductivity and high EMI shielding effectiveness to insulating polymers. This standard plating bath utilizes sodium hypophosphite as the reducing agent. **Figure 1.** Following the pre-treatment, the samples were immersed in the plating bath at 60 °C for 30 min. During processing, Nickel Chloride ($NiCl_2 . 6H_2O$): It is a source of nickel ions (Ni2+). Sodium Citrate ($C_6H5Na_3O_7 .2H_2O$): Functions as a complexing agent. It prevents nickel ions from reacting with hypophosphite, thereby inhibiting their precipitation as nickel phosphite within the solution. Ammonia (NH3.H2O): Functions as a buffering agent and an auxiliary complexing agent. It maintains the pH stability of the bath by neutralizing the hydrogen ions released during the reaction. Sodium Hypophosphite ($NaH_2PO_2 . H_2O$): Functions as a reducing agent. As a result of the reactions occurring within this bath, a Nickel-Phosphorus (Ni-P) alloy is deposited rather than pure nickel, exhibiting significantly superior properties in terms of electromagnetic shielding and corrosion resistance. Main Reduction Reaction is as foolow: [5]

$$H_2O + H_2PO_2- = H_2PO_3- + 2H \quad (2)$$

$$Ni_2+ + 2H = 2H+ + Ni \quad (3)$$

$$H_2PO_2- + H + H+ = P + 2H_2O \quad (4)$$

$$2H= H_2 \quad (5)$$

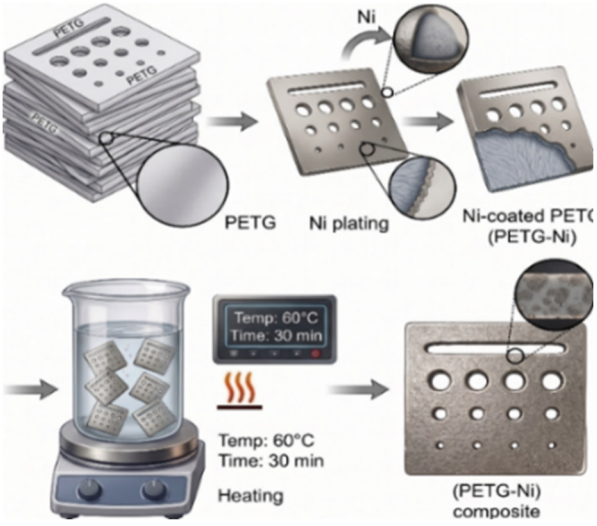

Figure 1 Schematic illustration of the electroless nickel plating procedure on 3D-printed PETG substrates to fabricate PETG-Ni composites.

2.3 Materials Test And Characterization

Following the aluminizing treatment, cross-sectional specimens were prepared using standard metallographic procedures for structural and microstructural characterization. The analyses were conducted using a JEOL JSM-5600 scanning electron microscope (SEM). Phase identification of the aluminide coatings was performed by X-ray diffraction (XRD) using a Rigaku X-ray diffractometer with Cu Kα radiation (λ = 1.5418 Å) over a 2θ range of 10°–90°. Elemental composition and phase stoichiometry were evaluated by SEM-EDS analyses, and coating thicknesses were measured from three different regions of the SEM micrographs. EMI SE is basically the practice of attenuating the electromagnetic wave using the proper shielding materials. Since the shielding material is a barrier between the signal source and the receiver, the shielding effectiveness is

associated with the insertion loss, which indicates the attenuation magnitude. Several test standards such as IEEE Std 299.1, MIL-STD-285, ASTM D4935, ASTM ES7-83 have been common evaluating shielding effectiveness.

3. RESULT AND DISCUSSION

As clearly illustrated in (**Figure 2a**), both PLA and PETG substrates exhibit a highly homogeneous and continuous nickel coating across their entire surfaces. The electroless deposition process successfully achieved full coverage, resulting in a uniform metallic layer that conforms to the underlying polymer topography without any observable discontinuities or uncoated regions. However, high-magnification SEM analysis reveals distinct morphological differences between the two polymers in terms of grain size and nucleation density. The Ni deposits on the PLA surface are characterized by significantly smaller, refined nodules that grow by overlapping and interconnecting with one another. This compact structure suggests a higher nucleation rate on the PLA, which facilitates a denser interfacial contact. Consequently, this refined morphology is expected to enhance the mechanical interlocking between the Ni layer and the PLA matrix, potentially leading to superior adhesion strength.

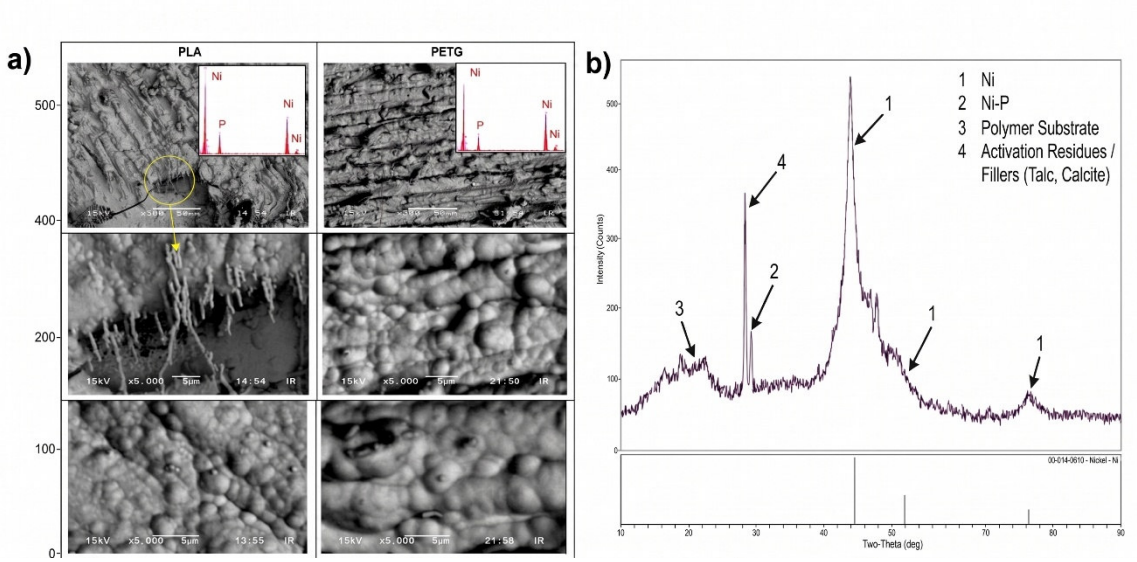

Figure 2 a) SEM microstructures and EDS spectrum of electroless Nickel coated PLA and PETG polymers with different magnifations. b) XRD diffraction patterns

In contrast, the PETG substrate displays a coarser morphology with larger, more isolated "cauliflower-like" grains. The increased grain size on PETG indicates a lower nucleation to growth ratio, where the Ni clusters grow more extensively before establishing a continuous film. While this structure provides substantial surface roughness, the relatively larger grain boundaries and less integrated overlap compared to PLA may influence the localized stress The EDS analysis further corroborates the SEM findings, confirming that the entire surface of both polymers is successfully covered with Nickel (Ni). As shown in the EDS spectra, the predominant peaks are attributed to Ni, providing chemical evidence for the formation of a continuous metallic layer across the substrates. The presence of Phosphorus (P) detected in the analysis is an inherent result of the production process, specifically originating from the chemical reagents used during the electroless deposition. This elemental distribution confirms that the coating process effectively metallized both PLA and PETG surfaces, with Ni being the primary deposited element distribution and the overall interfacial bonding characteristics.

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The XRD pattern (**Figure 2b**) corroborates the SEM/EDS findings. Peak 3 represents the amorphous PLA and PETG substrates, while peak 4 (~29°) originates from filament fillers or residual Sn/Pd catalysts. Peak 2 indicates localized crystalline Ni-P intermediate phases. Most notably, peaks marked 1 correspond to face-centered cubic (FCC) nickel. The significant broadening of the primary (111) peak at 44.5° confirms a nano-crystalline or nearly amorphous structure due to phosphorus incorporation. This specific Ni-P lattice structure directly explains the continuous nodular and "cauliflower-like" morphologies observed in the SEM images.

The total electromagnetic interference (EMI) shielding effectiveness of a material consists of reflection, absorption, and multiple reflections. If the absorption component exceeds 10 dB, multiple reflections can be neglected. Shielding performance was evaluated in the X-band (8.2–12.4 GHz) using a Vector Network Analyzer. As shown in (**Figure 3**), the metallized polymer samples demonstrate excellent attenuation, maintaining high total shielding effectiveness across the entire frequency range.

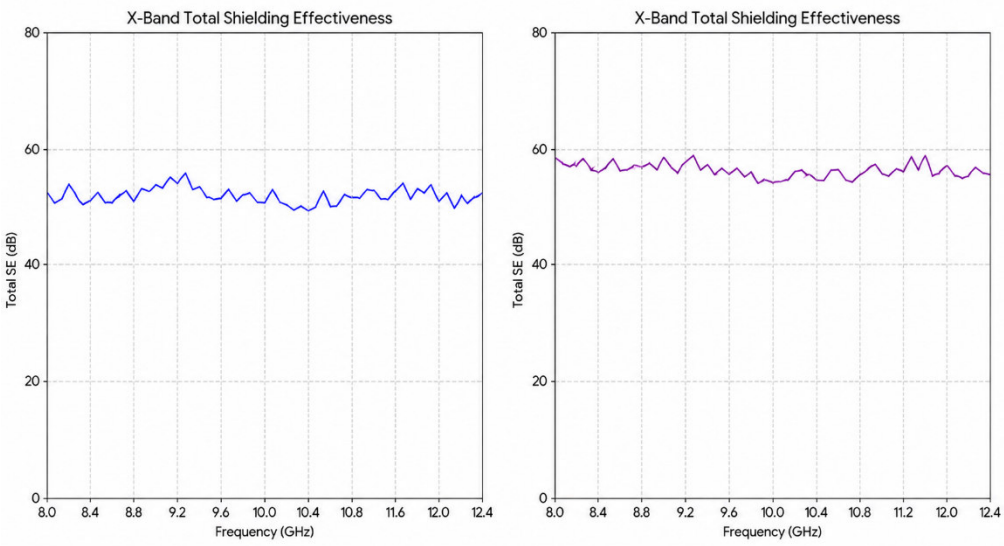

Figure 3 X-Band Total EMI Shielding Effectiveness (SE) of the metallized PLA and PETG polymer samples, respectively

4. CONCLUSION

Electromagnetic interference (EMI) poses a significant challenge to modern electronics, necessitating the development of lightweight and effective shielding alternatives to traditional metals. In this study, 3D-printed Polylactic Acid (PLA) and Polyethylene Terephthalate Glycol (PETG) polymers were successfully metallized using an electroless plating process to enhance their EMI shielding effectiveness (SE). Following proper surface activation with tin(II) chloride and palladium, continuous conductive networks were deposited, with PETG and PLA coated in nickel.

The overall electromagnetic shielding effectiveness of the materials is associated with three fundamental mechanisms: reflection, absorption, and multiple internal reflections. Reflection mainly arises from the impedance mismatch at the material–air interface, whereas absorption results from the attenuation and dissipation of electromagnetic energy as the wave propagates through the material. In this study, the shielding effectiveness was experimentally evaluated using a vector network analyzer (VNA) with waveguide flanges.

Evaluations in the X-band (8.2–12.4 GHz) demonstrated that the metallized polymer composites achieved a high total shielding effectiveness of up to ~60 dB. These results indicate that electroless-metallized 3D-printed polymers offer lightweight and structurally adaptable EMI shielding solutions with high shielding effectiveness.

ACKNOWLEDGEMENTS

This work was supported by the Sakarya University Scientific Research Projects (BAP) under Project No. 2026-29-85- 101.

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