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Microwave Absorbing Properties of Epoxy-SiO_2-Fe_3O_4 Hybrid Coatings on Plasma Electrolytic Oxidation-Treated Aluminum 6061

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Abstract

The escalation of radar detection technology has driven an urgent need for microwave-absorbing materials in stealth technology applications. This study investigates the microwave absorption capabilities of an Epoxy-SiO_2-Fe_3O_4 hybrid composite coating applied to Aluminum 6061 substrates treated with Plasma Electrolytic Oxidation (PEO). The incorporation of 5 g/L malonic acid during the PEO process produced an oxide base layer with a thickness of 5.14 ± 0.89 µm, featuring microporous characteristics that facilitate a mechanical interlocking mechanism for the composite layer. Variations in functional filler compositions (S, F, SF1, SF2, and SF3) were exclusively tested using a Vector Network Analyzer (VNA) across the X-band frequency range (8 - 12 GHz). The results indicated that all samples exhibited resonance peaks within the 8.7 - 9.26 GHz range. The most significant absorption was achieved by sample S (100% SiO_2) with a Reflection Loss (RL) value of -2.34 dB at 9.26 GHz, followed by sample SF3 (75% SiO_2 : 25% Fe_3O_4) at -1.91 dB at 9.02 GHz. This performance demonstrates the dominance of the dielectric loss mechanism at high frequencies, while the addition of Fe_3O_4 plays a strategic role in modifying magnetic permeability to optimize impedance matching. Although the RL values have not yet reached the technical threshold of -10 dB due to single-coat thickness limitations, the integration of PEO and functional hybrid layers successfully reduced microwave reflection intensity systematically on conductive metal surfaces.

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Abstrak

Eskalasi teknologi radar memicu kebutuhan mendesak akan material penyerap gelombang mikro untuk aplikasi teknologi siluman. Penelitian ini menginvestigasi kemampuan penyerapan gelombang mikro pada lapisan hybrid *komposit Epoxy-SiO_2-Fe_3O_4 yang diaplikasikan di atas substrat Aluminium 6061 hasil proses* Plasma Electrolytic Oxidation *(PEO). Penggunaan aditif asam malonat 5 g/L pada proses PEO menghasilkan* base layer *oksida setebal* 5,14 ± 0,89 *µm dengan karakteristik mikropori yang mendukung mekanisme* mechanical interlocking *bagi lapisan komposit. Variasi komposisi* filler *fungsional (S, F, SF1, SF2, SF3) diuji secara eksklusif menggunakan* Vector Network Analyzer *(VNA) pada rentang frekuensi* X-band *(8 - 12 GHz). Hasil menunjukkan bahwa seluruh sampel memiliki puncak resonansi pada rentang 8.7 – 9.26 GHz. Penyerapan paling signifikan dicapai oleh sampel S (100% SiO_2) dengan nilai* Reflection Loss *(RL) sebesar -2,34 dB pada 9,26 GHz, diikuti oleh sampel SF3 (75% SiO_2 : 25% Fe_3O_4) sebesar -1.91 dB pada 9.02 GHz. Performa ini membuktikan dominasi mekanisme* dielectric loss *pada frekuensi tinggi, sementara penambahan Fe_3O_4 berperan strategis dalam memodifikasi permeabilitas magnetik untuk optimalisasi* impedance matching. *Meskipun nilai RL belum mencapai ambang batas teknis -10 dB karena keterbatasan ketebalan lapisan tunggal, integrasi PEO dan lapisan* hybrid *fungsional ini berhasil mereduksi intensitas pantulan gelombang mikro secara sistematis pada permukaan logam konduktif.*

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1. Introduction

The escalation of detection technologies in modern defense systems, particularly through radar advancements, has triggered an urgent demand for the development of materials with stealth capabilities (Tang et al., 2021). Aluminum 6061 alloy is a material extensively implemented in structural components of military equipment and aerospace applications due to its superior strength-to-weight ratio and mechanical properties (Tham et al., 2007). However, as a conductive metal, aluminum possesses a high Radar Cross Section (RCS), causing electromagnetic waves that strike its surface to be reflected with high intensity toward the radar receiver. This reflection phenomenon makes the material highly susceptible to detection by opposing systems. Consequently, surface engineering through the application of Radar Absorbing Materials (RAM) has become a crucial solution to minimize these reflections by converting microwave energy into thermal energy within the coating structure. Enhancing this absorption capability serves not only as passive protection but also determines the operational effectiveness of defense equipment in complex combat environments (Kim et al., 2023).

In recent decades, the development of Radar Absorbing Materials (RAM) has explored various synthesis methodologies to optimize electromagnetic wave interactions through the modification of nanocomposite structures. Several commonly used approaches include the co-precipitation method, where iron salt ions are mixed with ammonia as a base to synthesize Fe_3O_4 nanoparticles with high magnetic response. Additionally, the Ultrasonic Spray Pyrolysis (USP) technique is frequently adopted for coating nanoparticles with noble metals to protect the magnetic core from oxidation and corrosion while enhancing chemical stability (Kresnik et al., 2024). Another popular method in the fabrication of carbon-based materials is the modified Hummers method, utilized to synthesize Graphene Oxide (GO) or reduced Graphene Oxide (rGO) from natural waste, such as coconut shells, due to its faster and safer oxidation efficiency compared to conventional methods (Triya et al., 2025). Despite various synthesis techniques being capable of producing materials with superior magnetic and dielectric characteristics, the primary challenge remains the significant tendency of particles to undergo agglomeration due to magnetic dipole-dipole attractions and low surface charges. This condition is exacerbated in conventional coating methods, such as painting or the use of adhesive stickers on conductive Aluminum 6061 metallic substrates, which often exhibit low adhesion strength and susceptibility to delamination when exposed to extreme environmental conditions. Consequently, such structural instabilities can hinder microwave absorption effectiveness due to non-uniform filler distribution and the loss of coating integrity at the substrate interface (Hariyawan et al., 2024).

As an effort to mitigate this material vulnerability, the Plasma Electrolytic Oxidation (PEO) technique was selected due to its capability to transform metal surfaces into ceramic oxide layers that maintain a robust metallurgical bond with the substrate (Lu, 2017). Based on previous studies, the optimization of the PEO process on Aluminum 6061 alloy using 5 g/L of malonic acid (MA) organic additive has been proven to significantly modify plasma discharge behavior, reduce the breakdown voltage, and produce high-density oxide layers. The incorporation of this additive is capable of producing layers with an average thickness $5.14 \pm 0.89\,\mu m$, which are structurally superior and denser compared to additive-free layers, which only reach an average thickness of $2.37 \pm 0.76\,\mu m$ (Lubis et al., 2025).

The natural microporous characteristics formed during the plasma oxidation process are utilized as a base layer to create a mechanical interlocking mechanism (Hosseini Rad et al., 2019). When the epoxy resin-based hybrid coating containing SiO_2 and Fe_3O_4 fillers is applied, the liquid phase of the resin penetrates these micropores before the polymerization process is complete. This mechanism creates exceptionally strong interfacial adhesion to prevent delamination while ensuring the stable distribution of magnetic and dielectric materials (Rodriguez et al., 2023). The primary novelty of this research lies in the integration of these functional hybrid layers to optimize dielectric loss through SiO_2 powder and magnetic loss through magnetite (Fe_3O_4). The synergy of these two materials within the epoxy matrix aims to minimize wave reflections at the air-material interface by adjusting its intrinsic impedance profile (Waryah et al., 2023).

This study specifically focuses on investigating the effect of varying the mass ratio between SiO_2 and Fe_3O_4—categorized into samples S, F, SF1, SF2, and SF3—on microwave absorption capabilities across the operational frequency range. Functional characterization was performed exclusively using a Vector Network Analyzer (VNA) to obtain scattering parameters, or S-parameters (S_{11}). Through the analysis of Reflection Loss (RL) derived from the VNA data, this research aims to identify the most optimal hybrid coating composition for achieving maximum microwave absorption. The ultimate objective of this study is to determine the compositional equilibrium point capable of producing minimum RL values below the technical threshold, thereby providing a significant contribution to the development of stealth material technology for future defense applications.

2. Research Methods

2.1 Materials and Experimental Equipment

The equipment utilized in this research includes a grinder-polisher, hot plate magnetic stirrer, ultrasonic cleaner, graduated cylinders, digital timers, recording devices, stirring rods, alligator clips, digital analytical balances, a DC power supply, a water cooling system, digital calipers, beakers, glass stirrers, and spatulas.

The materials consist of Aluminum 6061 alloy, distilled water (aquades), silicon carbide (SiC) abrasive paper, Potassium Hydroxide (KOH), Sodium Silicate (Na_2SiO_3), malonic acid ($C_3H_4O_4$), epoxy resin, hardener, silica (SiO_2), graphite plates, magnetite powder (Fe_3O_4), acetone, and ethyl alcohol.

2.2 Substrate Preparation

The initial phase begins with the preparation of the primary material, Aluminum 6061 alloy plates, to be used as the research substrate. The Aluminum 6061 plates are cut to specified dimensions of 3 x 4 cm to ensure a uniform surface area for all tested samples. Following the cutting process, the substrates undergo surface refinement using a grinding machine. This process is conducted incrementally using silicon carbide abrasive paper (sandpaper) with increasing grit sizes—specifically 240, 480, 600, 800, and 1000. The objective of this sequential grinding is to remove existing oxide layers and achieve optimal surface flatness. The final stage of preparation involves removing residual contaminants or greases resulting from the grinding process. The substrates are cleaned using an ultrasonic cleaner with a sequential immersion in acetone, ethanol, and distilled water (aquadest), for 5 minutes each. This cleaning procedure ensures that the substrate surface is completely sterile and prepared for the subsequent experimental stages.

2.3 Preparation of Electrolyte Solution

The preparation of the electrolyte solution in this study was conducted by mixing several primary chemical reagents according to predetermined compositions. The first stage involved preparing 1 liter of distilled water (aquadest) as the solvent, which was then mixed with 6.172 g of Potassium Hydroxide (KOH) and 15 g of Sodium Silicate (Na_2SiO_3) as the base electrolyte components. Once the base solution reached homogeneity, 5 g of malonic acid additive was introduced into the mixture. All materials were subsequently stirred thoroughly to ensure complete dissolution of all particles, rendering the electrolyte solution ready for the next experimental phase.

2.4 PEO Experimental Set-up

The equipment assembly utilized to support the plasma oxidation process on the specimen surface was systematically arranged to ensure the stability of electrical and thermal parameters. This experimental installation integrates a DC power source with an electrolysis system equipped with temperature control and solution homogeneity management. The detailed device configuration and the placement of key components in the Plasma Electrolytic Oxidation (PEO) process are shown schematically in **Figure 1**.

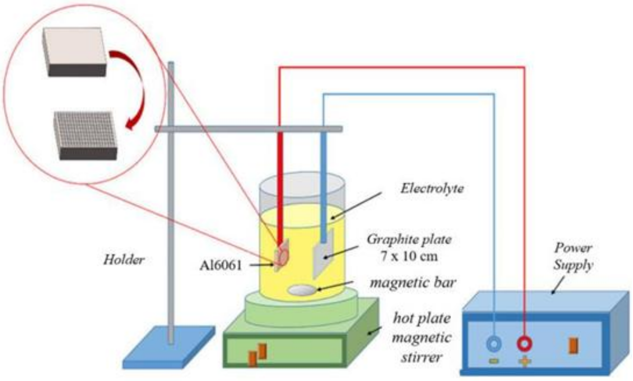

Figure 1. Schematic diagram of the Plasma Electrolytic Oxidation (PEO) experimental setup.

The PEO coating process was conducted by configuring the Aluminum 6061 substrate as the anode and a 7 x 10 cm graphite plate as the cathode within a glass vessel containing the electrolyte solution. The system was connected to a Programmable DC Power Supply using high-conductivity connectors to supply the voltage required to trigger plasma discharges on the specimen surface. The current density was regulated based on the total surface area of the substrate to ensure uniform layer distribution during the 4-minute oxidation duration. To maintain solution homogeneity and electrolyte temperature stability below 25°C, a combination of a magnetic stirrer and an external cooling circulation system was employed. Precise temperature regulation is critical to maintaining micro-plasma stability and preventing thermal damage to the developing oxide layer.

2.5 Application of Functional Composite Coating

Following the PEO process, the next stage involves applying a functional composite coating to the specimen surface. This process begins with the preparation of the coating material, utilizing epoxy resin and hardener as the binding matrix, with the addition of silica (SiO_2) and magnetite (Fe_3O_4) as functional fillers. All materials are mixed in a beaker glass using a stirring rod with specific mass proportions to produce five sample categories: S (100% SiO_2), F (100% Fe_3O_4), SF1 (25% SiO_2 : 75% Fe_3O_4), SF2 (50% SiO_2 : 50% Fe_3O_4), and SF3 (75% SiO_2 : 25% Fe_3O_4).

To ensure mixture homogeneity, the SiO_2 and Fe_3O_4 particles dispersed within the epoxy matrix are processed using ultrasonication for 5 minutes. Once the optimal dispersion level is achieved, the mixture is applied directly onto the PEO-treated surface through a single-coat application. This single-layer method is intentionally employed to maximize the utilization of the PEO-induced microporous structure as sites for mechanical interlocking before the resin polymerization is complete. This procedure guarantees the formation of a homogeneous single layer with robust interfacial bonding, ensuring the stable distribution of magnetic and dielectric materials for subsequent microwave absorption characterization using a VNA.

2.6 Sample Characterization

The functional characteristics of the fabricated composite coatings were analyzed exclusively using a Vector Network Analyzer (VNA). Measurements were conducted across the X-band frequency range (8–12 GHz) to evaluate the material's interaction with microwaves. The primary parameter measured was the reflection coefficient, or scattering parameter (S_{11}), which represents the logarithmic ratio between the amplitude of the wave reflected by the specimen surface and the amplitude of the incident wave.

The processing of VNA data focuses on determining the Reflection Loss (RL) values, expressed in decibels (dB). Mathematically, the RL value is derived from the S_{11} data to determine the electromagnetic energy dissipation efficiency of the material. Analysis was conducted by plotting the relationship between RL and frequency to identify the matching frequency (resonance frequency) where absorption reaches its maximum point. Furthermore, this data processing aims to evaluate the influence of varying magnetic and dielectric filler compositions on the absorption peak shift and the effective absorption bandwidth, which serve as primary indicators of the material's performance in reducing microwave reflection intensity on metal surfaces. A more negative RL value indicates a higher capability of the material to convert radar wave energy into other forms of energy, specifically heat. Materials with an RL value lower than -10 dB are generally considered effective for microwave absorption, as this represents 90% energy absorption (Gunanto et al., 2022).

To understand the absorption phenomena observed in the VNA data, the wave interaction mechanism within this dual-coating system is schematically illustrated in **Figure 2**.

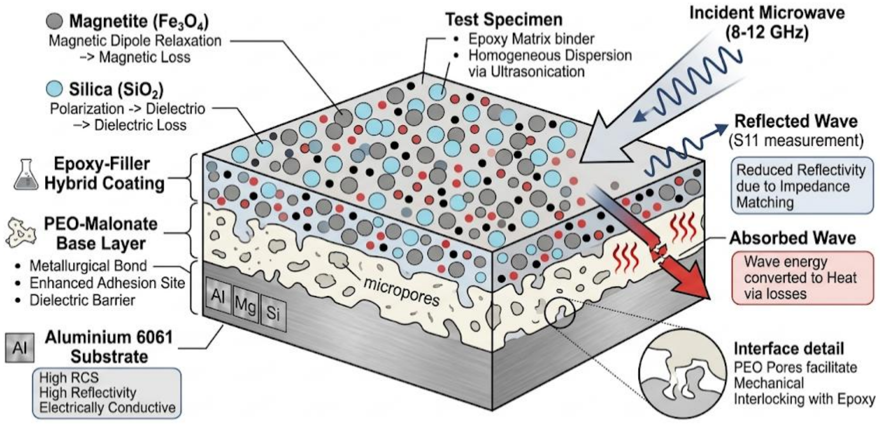

Figure 2. Schematic of electromagnetic wave interaction and attenuation mechanisms in the PEO-composite coating system (Generate by Gemini).

Phenomenologically, the wave attenuation mechanism within this system involves a synergy between impedance matching conditions and the efficiency of energy dissipation within the functional composite matrix, as illustrated in Figure 2. The adjustment of filler composition plays a vital role in aligning the material's intrinsic impedance with the impedance of free space. This alignment ensures that electromagnetic waves can propagate into the coating structure with minimal surface reflection. Subsequently, the attenuation process occurs through dielectric loss from the silica and magnetic loss from the magnetite, which cooperatively convert electromagnetic energy into thermal energy (X. Chen et al., 2021).

The observed absorption effectiveness is inseparable from the interfacial integrity formed by the PEO layer, optimized with 5 g/L of malonic acid additive. According to Lubis et al. (2025), this ceramic oxide layer, with a thickness of $5.14 \pm 0.89\ \mu m$, serves not only as an additional electrical insulator for the Aluminum 6061 substrate but also provides a crucial morphological foundation for the composite layer (Lubis et al., 2025). The fine microporous structure resulting from the PEO process facilitates a robust mechanical interlocking mechanism, in which the epoxy resin matrix penetrates these pores before the polymerization process is finalized (Hosseini Rad et al., 2019). This ensures the stable distribution of the SiO_2 and Fe_3O_4 functional fillers across the substrate surface. Furthermore, the strong interfacial adhesion minimizes electromagnetic discontinuities that could lead to undesired wave scattering, thereby stabilizing the energy dissipation process (Pavarini et al., 2025). Consequently, the integration of a dense, thick PEO layer with a functional hybrid coating creates a dual-protection system that combines material durability with effective wave attenuation capabilities.

3. Results and Discussions

In the initial stage, a visual observation was conducted on the hybrid coatings applied to the PEO-treated Al6061 substrates. **Figure 3** illustrates the physical appearance of the specimens for each compositional variation.


Sample S Sample F Sample SF1 Sample SF2 Sample SF3

Figure 3. Physical documentation of the hybrid composite-coated specimens

Visually, a distinct difference in surface color gradation is observed, where samples containing Fe_3O_4 (Sample F) exhibit a darker hue compared to the sample with pure SiO_2 (Sample S). This indicates that the functional fillers have been successfully dispersed within the epoxy matrix and are uniformly adhered to the PEO base layer surface (Ma et al., 2016).

The evaluation of electromagnetic wave absorption capabilities of the coated specimens was conducted by measuring the S_{11} scattering parameters using a Vector Network Analyzer **(VNA)**. The measurement results are presented in **Figure 4** and summarized in **Table 1**.

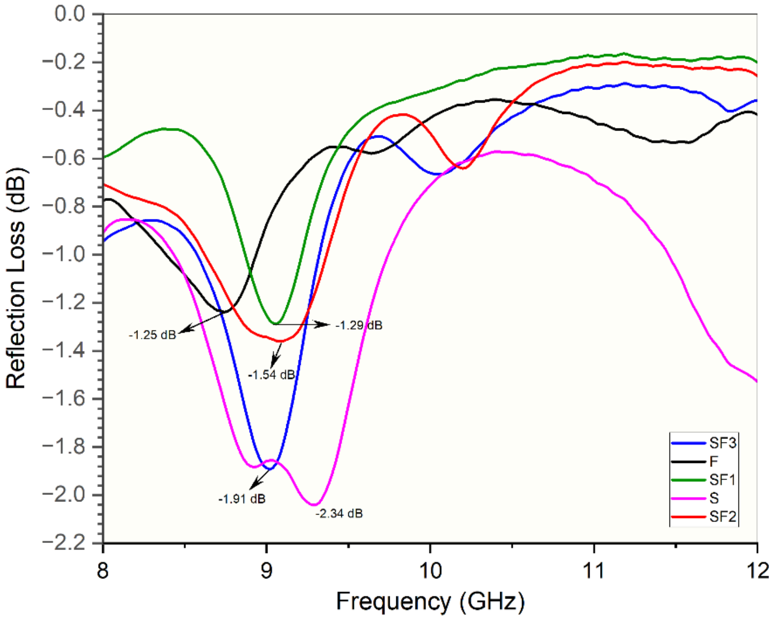

Figure 4. Reflection Loss vs. Frequency graph for all sample variations

Table 1. Electromagnetic wave absorption performance across various filler composition variations

Sample	Composition (*Filler*)	*Matching Frequency* (GHz)	*Reflection Loss* (dB)
S	100% SiO_2	9.26	- 2.34
F	100% Fe_3O_4	8.7	- 1.25
SF1	25% SiO_2 : 75% Fe_3O_4	9.04	- 1.29
SF2	50% SiO_2 : 50% Fe_3O_4	9.14	- 1.54
SF3	75% SiO_2 :25% Fe_3O_4	9.02	- 1.91

The reflection coefficient data were converted into Reflection Loss (RL) values to determine the dissipative efficiency of the material within the X-band frequency range. All sample variations exhibited dominant resonance peaks (matching frequencies) localized between 8.7 GHz and 9.26 GHz. The occurrence of a matching frequency within a specific range is a physical phenomenon triggered by destructive interference between waves reflected from the coating surface and those reflected from the metal substrate interface (Syahputra et al., 2024).

Based on **Table 1**, the most significant absorption was achieved by Sample S (100% SiO_2), with a Reflection Loss (RL) value of -2.34 dB at a frequency of 9.26 GHz. The superior performance of Sample S and the hybrid mixture SF3 indicates that within the X-band frequency range, the dielectric polarization mechanism provides a more dominant contribution to absorption compared to the magnetic mechanism. Nevertheless, the observed shift in peak frequency among the hybrid samples (SF1, SF2, SF3) demonstrates the strategic role of Fe_3O_4 in modifying the magnetic permeability of the layer to support overall absorption effectiveness (Triya et al., 2025). Specifically, Fe_3O_4 provides Reflection Loss through the magnetic loss mechanism, where its ferrimagnetic properties and large magnetic anisotropy allow the material to absorb energy from the magnetic field component of the incident wave and convert it into thermal energy through magnetic dipole relaxation. This absorption efficiency is also closely linked to the morphological foundation of the PEO layer, optimized with malonate additives as shown in **Figure 3**, where the PEO microporous structure plays a vital role in ensuring the stable distribution of functional fillers over the reflective aluminum surface (Lubis et al., 2025).

The addition of Fe_3O_4 (F) in this hybrid system theoretically aims to optimize the magnetic loss mechanism through high magnetic response to broaden the absorption bandwidth. In this system, SiO_2 plays a crucial role in increasing the Reflection Loss value when combined with Fe_3O_4 by regulating the material's intrinsic impedance profile to align with the impedance of free space (impedance matching). The presence of silica as a dielectric component facilitates radar waves to propagate into the coating structure with minimal surface reflection, allowing Fe_3O_4 particles to dissipate electromagnetic energy more effectively within the composite matrix (Triya et al., 2025).

However, observations from the VNA plots indicate that the Reflection Loss (RL) values tend to decrease (weakened absorption) as the Fe_3O_4 concentration increases. This performance degradation phenomenon can be explained by the imbalance between dielectric and magnetic losses in thin single-coat applications. An increase in Fe_3O_4 mass without a corresponding increase in material thickness to reach its critical value can lead to impedance mismatch. In this condition, high magnetic properties actually increase the surface reflectivity of the material before the waves can propagate into the coating structure to be absorbed (Kresnik et al., 2024)(Syahputra et al., 2024). Material thickness significantly influences microwave absorption, where thicker layers are required to shift the matching frequency lower and maximize absorption (Hariyawan et al, 2024).

In addition to thickness factors, low mixture stability also triggers the agglomeration or clumping of Fe_3O_4 particles. Without an optimal dispersing agent, the natural magnetic attraction between particles causes the magnetite to stick together and form large clusters. This condition is characterized by a low zeta potential value, indicating that the particles lack sufficient repulsive forces to remain uniformly dispersed. To prevent such cluster formation, necessary strategies include the use of dispersing agents, such as polyvinylpyrrolidone (PVP), to create steric hindrance, and the optimization of high-energy sonication processes to break down particle clusters prior to the coating process. Consequently, if these preventive measures are not implemented, the clusters will disrupt the impedance matching because radar waves will be immediately reflected at the cluster surfaces before they can penetrate the layer to be absorbed and converted into thermal energy (Kresnik et al., 2024).

The absorption performance achieved in this study demonstrates a significant influence of the synthesis method when compared to previous research. As a direct comparison, the study by Lubis et al. (2025), which utilized identical PEO process parameters and malonic acid additives but without the addition of a functional hybrid layer, only yielded an RL value of -1.279 dB. The improvement of the RL value to -2.34 dB in this study proves that the integration of the Epoxy-SiO_2-Fe_3O_4 hybrid coating method after the PEO process provides a systematic additional contribution to reducing microwave reflection intensity through a dual attenuation mechanism (dielectric and magnetic) (Lubis et al., 2025). However, when compared to other synthesis methods, such as the use of Fe_3O_4 and Graphene Oxide (GO) nanocomposites processed using a ball mill at 300 rpm, significantly higher absorption efficiency can be achieved. Research by Syahputra et al. (2024) showed that a composition of 40% Fe_3O_4 and 60% GO was capable of producing a maximum absorption coefficient of 96.43% at a frequency of 8.42 GHz, with an RL value reaching -28.95 dB. This contrasting performance indicates that while the PEO method excels in creating strong metallurgical bonds and mechanical interlocking at the substrate interface, the optimization of RL values is highly dependent on the homogeneity of filler dispersion and the attainment of the material's critical thickness. Material thickness significantly influences absorption, where thicker layers can lower the matching frequency and increase the absorption capacity beyond the technical threshold required for practical applications (Syahputra et al., 2024).

Although the RL values in this study have not yet reached the ideal threshold of -10 dB (representing 90% absorption) required for practical applications, the incorporation of Fe_3O_4 remains an efficient approach for Radar Absorbing Material (RAM) components. The addition of Fe_3O_4 has been proven to facilitate the systematic modification of absorption peak frequencies and enhance the material's adsorption capacity toward electromagnetic waves. The synergistic integration of the magnetic loss characteristics of Fe_3O_4 with the dielectric properties of SiO_2 in this research constitutes a valid solution for reducing microwave reflection intensity on conductive metallic surfaces.

4. Conclusions

This study successfully investigated the influence of varying mass ratios of SiO_2 and Fe_3O_4 on the radar absorption capabilities of Aluminum 6061 substrates integrated with a PEO layer. Characterization results using a VNA demonstrated that all sample variations (S, F, SF1, SF2, and SF3) possess the ability to reduce electromagnetic wave reflection intensity within the X-band frequency range. The most optimal composition for achieving maximum microwave absorption was found in Sample S (100% SiO_2), which exhibited the most significant minimum Reflection Loss value compared to the other samples. Although the addition of Fe_3O_4 theoretically aims to optimize impedance matching, the dominance of the dielectric characteristics of SiO_2 proved to be more effective in reaching the absorption equilibrium point under thin single-coat conditions. Nevertheless, the role of Fe_3O_4 remains strategic in modifying the magnetic permeability of the layer to support absorption effectiveness through impedance matching optimization. This integration proves the potential for developing a dual-protection system that combines material durability with wave attenuation capabilities, which is crucial for the advancement of radar-absorbing materials in future stealth technology applications.

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