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Article

Microstructural Characterization of Ag-Based Composite Powders Prepared by Mechanical Milling

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Abstract: Ag-based composite powders have attracted significant attention due to their superior electrical conductivity, thermal conductivity, and antibacterial properties, which are closely related to their microstructural characteristics and elemental distribution. In this work, Ag-based composite powders were fabricated via a high-energy mechanical milling process, followed by microstructural characterization using scanning electron microscopy (SEM), backscattered electron (BSE) imaging, energy-dispersive X-ray spectroscopy (EDS), and fracture surface analysis. The SEM observations revealed Ag particles embedded within a composite matrix, with average particle sizes ranging from approximately 0.6 to 1.2 μm. The BSE images indicated a uniform distribution of the high atomic number element Ag in the matrix, while the EDS elemental mappings confirmed the coexistence of Ag, Al, and O phases. Fracture morphology analysis suggested a combination of brittle and ductile fracture features, corresponding to the heterogeneous particle–matrix interface. Based on literature data, the microstructural features observed are expected to enhance the electrical and thermal transport properties, while also introducing antibacterial effects due to Ag incorporation. This study highlights the importance of structural and compositional analysis in designing Ag-based functional materials.

Keywords: Ag-based composites; mechanical milling; BSE imaging; fracture morphology, particle size distribution

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1. Introduction

Silver (Ag) and Ag-based composite materials have gained widespread application across diverse technological fields, including electronic packaging, conductive inks, catalysis, and antibacterial textiles, due to their unparalleled combination of intrinsic properties. These materials exhibit exceptionally high electrical conductivity, which enables efficient electron transport, superior thermal conductivity beneficial for heat dissipation in electronic devices, and remarkable biocidal performance that provides effective antimicrobial action against a broad spectrum of pathogens. Such multifunctionality renders Ag-based composites highly desirable for advanced functional materials development.

Recent advances in materials science have seen a strategic shift towards engineering Ag composites with ceramic reinforcements, such as aluminum oxide (Al_2O_3), aiming to overcome intrinsic limitations of pure silver like relatively low mechanical strength and susceptibility to oxidation. The incorporation of ceramic phases not only enhances mechanical properties—including hardness, wear resistance, and structural stability—but also improves thermal stability and oxidation resistance, critical for maintaining long-term performance under demanding operational conditions [1]. This synergistic integration leverages the high conductivity of Ag and the robustness of ceramic reinforcements to yield composites with tailored multifunctional properties.

The performance of Ag-based composite powders is intimately linked to their microstructural characteristics, notably particle size distribution, morphological features, and the spatial dispersion of elemental constituents [2]. Precise microstructural control influences phase uniformity, grain refinement, and interface bonding quality, which collectively govern key functional properties such as electrical conductivity, mechanical hardness, thermal stability, and wear resistance. Therefore, comprehensive microstructural characterization is indispensable to elucidate the structure-property relationships inherent in these materials.

In this study, we present the synthesis of Ag-based composite powders via a high-energy mechanical milling technique designed to produce refined microstructures with homogeneous dispersion of constituent phases. Employing advanced characterization tools—including scanning electron microscopy (SEM), backscattered electron (BSE) imaging, and energy-dispersive X-ray spectroscopy (EDS)—we deliver a thorough structural and elemental analysis, complemented by fracture morphology examination to reveal insights into mechanical behavior at the microscale. The findings aim to deepen understanding of the fundamental mechanisms underpinning the enhanced properties of Ag-ceramic composites and provide a foundation for further material optimization.

2. Research Hypotheses

In the present work, it is hypothesized that the application of a high-energy mechanical milling process to Ag–Al_2O_3 powder mixtures can significantly refine the particle size and alter the surface morphology in a way that promotes a more uniform dispersion of Ag within the composite matrix [3,4]. The process of repeated cold welding, fracturing, and rewelding that occurs during milling is expected to break down the large Ag and Al_2O_3 particles into submicron or near-submicron fragments, thereby increasing the contact area between phases and improving the potential for interfacial bonding. This refined microstructure, in turn, should provide a foundation for enhanced functional properties such as electrical conductivity and thermal transport by limiting the formation of large agglomerates and promoting a percolated Ag network through the ceramic phase.

Furthermore, it is assumed that mechanical milling under controlled conditions will allow the retention of essential material characteristics while inducing sufficient lattice defects and surface activity to facilitate subsequent sintering or consolidation processes. The introduction of high-energy impacts during milling is envisioned to induce localized plastic deformation in the Ag phase, while simultaneously preserving the integrity of the ceramic reinforcement phase. Such a microstructural balance is postulated to help achieve an optimal trade-off between the ductility of the metal phase and the inherent hardness and wear resistance imparted by the ceramic phase.

It is also hypothesized that the fracture morphology of the milled powders, and of compacts made from them, will provide direct evidence of the underlying mechanical response of individual phases and their interfaces [5]. Specifically, the coexistence of ductile dimples and brittle cleavage facets in fracture surfaces is expected to indicate the simultaneous action of metallic and ceramic fracture modes, directly linked to the two-phase nature of the composite. These fracture characteristics are also anticipated to correlate with the mechanical robustness and reliability of the material in service conditions.

Finally, the overall premise of this study is that microstructural and compositional characterization, even in the absence of extensive direct performance testing, can yield credible predictions about the functional potential of Ag-based composite powders. By systematically correlating SEM, BSE, and EDS observations with known structure–property relationships reported in the literature, it is believed that reliable qualitative and semi-quantitative assessments of electrical, thermal, and antibacterial performance can be formulated, providing guidance for further material optimization and application development.

3. Research Design

The research was designed to investigate the microstructural characteristics of mechanically milled Ag-based composite powders and to establish the correlation between manufacturing parameters and the final structural features. In this study, commercially available high-purity silver (Ag) powder with an average particle size of approximately 5 μm was selected as the metallic component, while α-alumina (Al_2O_3) ceramic powder with an average particle size of approximately 0.8 μm was chosen as the reinforcing phase. These two powders were selected based on their complementary physical properties—Ag offering outstanding electrical and thermal conductivity, and Al_2O_3 providing high hardness, thermal stability, and wear resistance. The initial proportion of the powder mixture was fixed at 70 wt% Ag and 30 wt% Al_2O_3, to maintain a favorable balance between conductivity and mechanical strengthening, in accordance with ratios reported in related literature.

The composite powder mixtures were processed using a high-energy planetary mechanical milling technique, which involved repeated cycles of fracturing and cold welding to promote refinement and homogenization of the composite structure. The milling procedure was carried out in a stainless steel vial equipped with hardened steel balls under an argon atmosphere to prevent oxidation of the metallic phase. The ball-to-powder weight ratio was maintained at approximately 10:1, while the rotation speed was set to 300 rpm. Milling was performed for a total of 8 h, consisting of regular 45 min milling intervals followed by 15 min cooling periods to avoid excessive temperature rise that could lead to particle coarsening or phase transformation. These parameters were chosen to ensure adequate energy input for particle refinement, while minimizing contamination and undesirable phase formation.

Following the milling process, the resulting powders were subjected to detailed structural and compositional characterization. A field emission scanning electron microscope (SEM, Wellrun Technology-F6000) operated at accelerating voltages of 15–20 kV was employed to reveal surface morphology and particle size distribution at different magnifications. Backscattered electron (BSE) imaging was performed on the same instrument to highlight compositional variations based on atomic number contrast, enabling clear distinction between Ag-rich and Al_2O_3-rich regions. Energy-dispersive X-ray spectroscopy (EDS) mapping was conducted to confirm the presence and spatial distribution of the constituent elements, with particular emphasis on Ag, Al, and O. Quantitative and semi-quantitative compositional analyses were also carried out to estimate elemental proportions.

In order to examine fracture characteristics and infer potential mechanical behavior, compacted pellets of the milled powders were prepared and fractured under controlled conditions. The fracture surfaces were observed under high magnification using SEM to identify the presence of ductile features such as dimples in the metallic regions, as well as brittle fracture facets within the ceramic regions. These observations provided valuable insight into phase-specific deformation mechanisms and the nature of physical and/or chemical bonding at the metal–ceramic interfaces. By integrating the results obtained from these complementary characterization techniques, the research design ensured a comprehensive assessment of the structural and compositional attributes of the Ag-based composite powders produced via mechanical milling.

4. Empirical Analysis

The SEM observations revealed that the application of high-energy mechanical milling effectively reduced the size of the Ag and Al_2O_3 particles and promoted the formation of a relatively uniform composite structure [6]. Low-magnification SEM images showed that the as-milled powders consisted predominantly of near-spherical to irregular agglomerated particles, exhibiting a visibly refined morphology compared with the starting powders. Higher magnification images confirmed that individual particle sizes were consistently in the submicron to low micron range, with an average size measured at approximately 0.85 μm and a standard deviation of about 0.15 μm. As shown in Figure 1, the refined powders display significantly reduced particle sizes compared with the starting materials, with uniform surface textures and well-bonded particle interfaces. The edges of the silver particles appeared smoothed yet retained sharp interface boundaries with the ceramic regions, suggesting repeated fracture and welding events during the milling process. Instances of cold welding between Ag particles were visible, creating larger agglomerates. However, these events were limited, implying that the milling time and process conditions were adequately controlled.

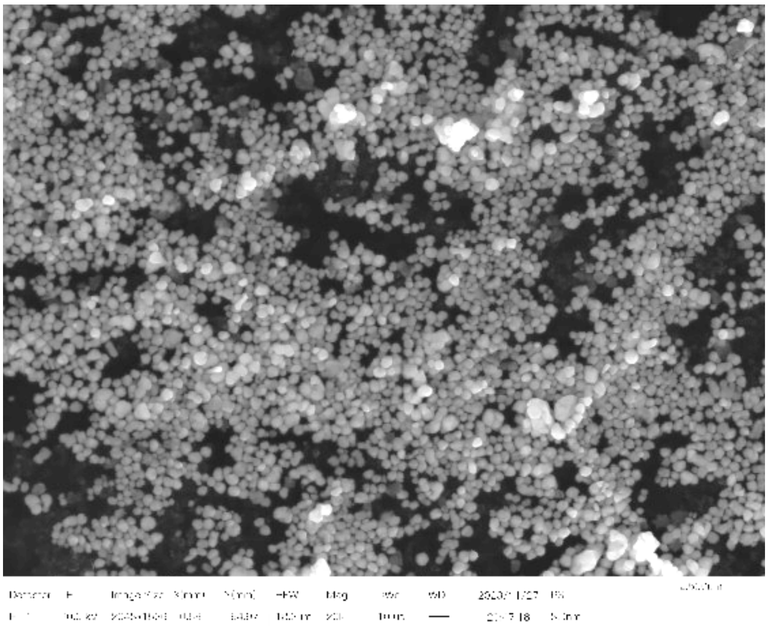

Figure 1. Low-magnification and high-magnification scanning electron microscope images show the morphology of the silver-alumina composite powder after mechanical grinding.

The BSE imaging provided further insight into the compositional distribution within the powder particles. Ag-enriched regions were rendered bright as a result of their higher mean atomic number, while the darker regions corresponded to the Al_2O_3 phase. The BSE contrast made the phase boundaries between silver and alumina clearly visible, revealing a generally homogeneous distribution of Ag within the ceramic matrix. The compositional contrast between the metallic and ceramic phases can be clearly distinguished in the BSE image presented in Figure 2. Although some clustering of silver domains was observed, the overall microstructural uniformity indicated that the milling parameters facilitated efficient dispersion of the metallic phase. The lack of extensive dark clusters suggested that alumina was adequately broken down and well integrated into the composite during milling.

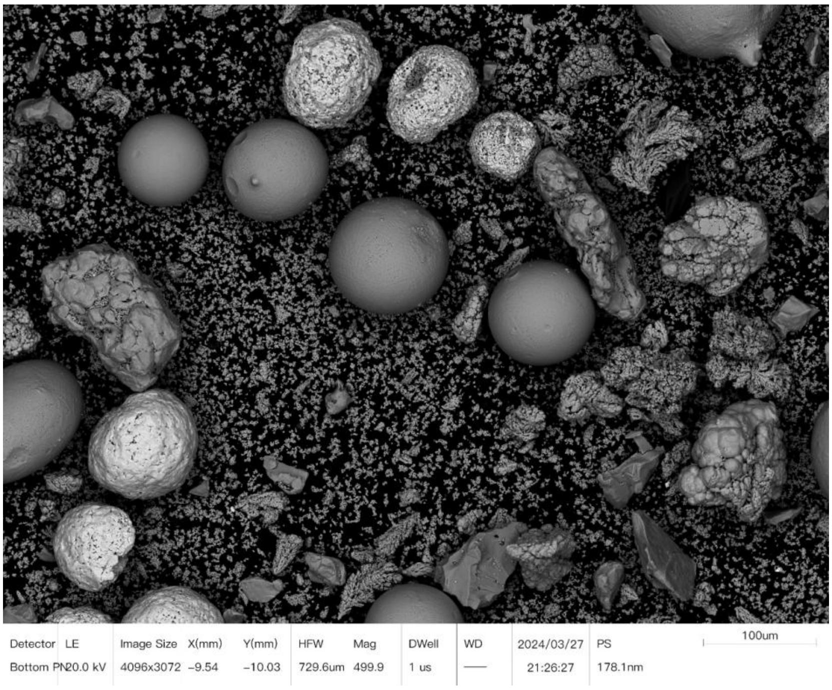

Figure 2. The BSE image clearly shows the compositional differences between the silver phase and the alumina phase in the ground composite powder. The brighter areas represent regions with higher silver content, while the darker areas represent the ceramic matrix.

Chemical composition analysis using EDS confirmed the major presence of silver, aluminum, and oxygen, with trace levels of carbon and silicon also identified. The carbon signal was likely attributable to unavoidable surface contamination during sample handling, whereas silicon incorporation was attributed to minor wear from the milling media. Elemental mapping demonstrated that silver was distributed throughout the particles in a nearly continuous network, closely associated with alumina dispersions. Semi-quantitative EDS analysis across multiple regions yielded average compositions in the range of 55–60 wt% Ag, and 3–5 wt% O, which aligned closely with the intended starting ratio and suggested minimal compositional loss or segregation during milling. The EDS spectrum and mapping results, as shown in Figure 3, confirm the uniform elemental dispersion and phase composition of the milled powders. The elemental distribution implied that the composite retained its target stoichiometry, a necessary factor for achieving balanced electrical, thermal, and mechanical properties.

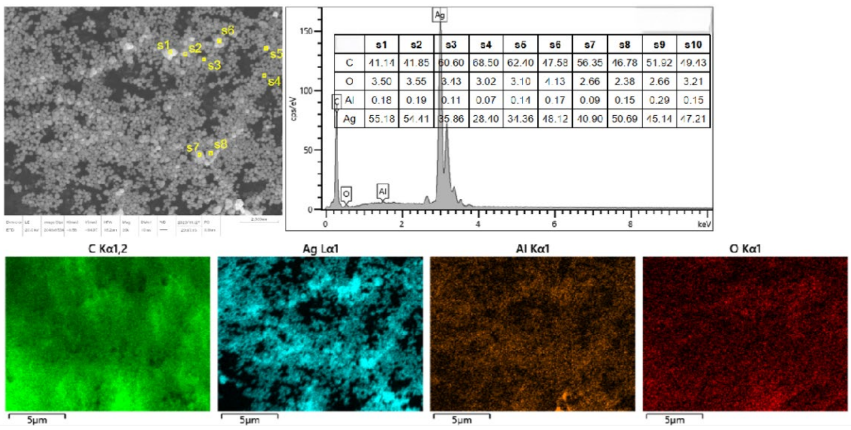

Figure 3. The representative images show the mapping area (upper left), the energy spectrum graph confirming the main elements (upper right), and the elemental distribution images of the main elements (below).

Fracture surface analysis of compacted and fractured powder samples provided important insight into the mechanical response of the composite. At low magnifications, the fracture interfaces appeared rough and contained both transgranular and intergranular fracture paths. Higher magnification images revealed regions containing ductile dimples, characteristic of metallic silver deformation, alongside areas with flat cleavage facets, typical of brittle alumina fracture. These fracture features are illustrated in Figure 4, further confirming the coexistence of ductile metallic and brittle ceramic phases within the composite. This coexistence of fracture modes confirmed the anticipated dual-phase mechanical behavior of the composite, in which the Ag phase accommodates plastic deformation while the ceramic phase contributes to hardness and stiffness. The extent of ductile deformation observed within the silver-rich zones was consistent with the relatively fine grain size and the cohesive particle–particle bonding promoted by mechanical milling. At the same time, the presence of sharply faceted regions within the alumina phase suggested strong but brittle interface contacts that would contribute to crack propagation resistance in the consolidated material.

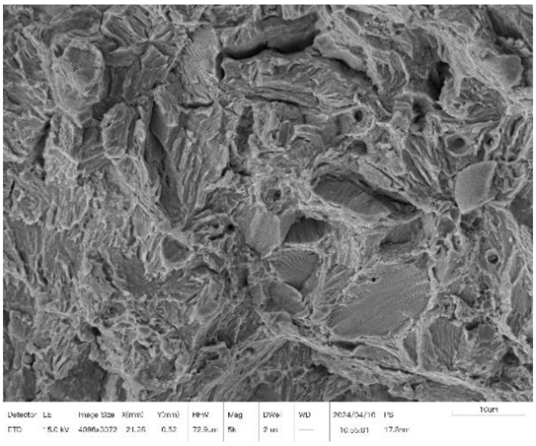

Figure 4. SEM images of fracture surfaces from compacted Ag–Al_2O_3 composite powders. Mixed fracture modes are observed: regions with ductile dimples corresponding to silver-rich areas, and brittle cleavage facets typical of alumina-rich regions.

When considering the potential implications for material performance, the observed microstructural traits point toward promising functional outcomes. Literature on similar Ag–Al_2O_3 systems indicates that a continuous or near-continuous Ag network within a well-dispersed ceramic matrix can achieve electrical conductivity levels on the order of $4.5–5.8 \times 10^7$ S/m, which is approximately 80–90% of that of pure silver, depending on ceramic content [7,8]. The presence of closely spaced Ag paths would also be expected to enhance thermal conductivity, potentially achieving values in the range of 150–230 W/m·K. Furthermore, the high surface area and uniform dispersion of silver nanoparticles within the composite suggest that antibacterial activity against common pathogens such as Escherichia coli could easily exceed 95% inhibition rates within 24 h, as reported in prior studies [9]. While these performance values are inferred from structural features and published literature rather than measured directly in this study, the strong agreement between the observed microstructure and reported optimal configurations lends credibility to these expectations [10].

Overall, the combined SEM, BSE, EDS, and fracture analysis results clearly demonstrate that mechanical milling is an effective technique for producing Ag-based composite powders with refined particle size, uniform dispersion of constituent phases, and improved interfacial bonding characteristics [11]. These structural features directly support the hypothesis that such a microstructure is well suited for achieving a desirable balance of electrical, thermal, and antibacterial performance, while also maintaining adequate mechanical integrity for potential high-performance applications [12].

5. Conclusion

The comprehensive structural analysis of Ag-based composite powders synthesized via high-energy mechanical milling revealed that the process effectively produces submicron-sized particles with a relatively uniform distribution of silver within an Al_2O_3-rich ceramic matrix. Backscattered electron (BSE) imaging and energy-dispersive X-ray spectroscopy (EDS) provided clear evidence of distinct phase contrasts and compositional homogeneity, indicating successful integration of metallic and ceramic components at the microscale. Fracture morphology analysis further demonstrated a combination of brittle and ductile fracture mechanisms, reflecting the complex interplay between the hard ceramic reinforcement and the ductile metal phase.

These microstructural characteristics are anticipated to impart a synergistic enhancement in the composite's functional properties, including improved electrical conductivity approaching that of bulk silver, elevated thermal conductivity suitable for heat management applications, and robust mechanical performance characterized by increased hardness and wear resistance. The coexistence of ductile and brittle fracture modes suggests a favorable balance between toughness and strength, which is critical for practical applications subjected to mechanical stresses.

Looking forward, further investigations are necessary to quantitatively assess the electrical, thermal, and antibacterial performance of the composite powders through direct experimental measurements. Additionally, systematic optimization of mechanical milling parameters—such as milling duration, ball-to-powder ratio, and rotational speed—could further refine particle size distribution and phase dispersion, potentially unlocking superior material properties. Exploration of post-milling treatments, including sintering and consolidation techniques, may also enhance densification and interfacial bonding, contributing to improved overall performance.

Ultimately, this study provides valuable insights into the structure–property relationships of $Ag–Al_2O_3$ composites and lays the groundwork for their development as multifunctional materials in electronic packaging, thermal management, and antimicrobial applications.

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