

Review

Preparation and Classification of Coatings by High-Energy Ball Milling: A Review

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Abstract

High-energy ball milling (HEBM) offers a pathway for the green preparation of multifunctional coatings. However, existing research lacks systematic frameworks addressing the interplay of HEBM process parameters, elemental screening criteria, and coating classification systems. This study establishes a comprehensive “elemental screening–process synergy–classification and prediction” framework for HEBM coatings. Key contributions include establishing a two-tier screening criterion based on non-radioactivity/low-toxicity and functionality for coating elements; revealing the synergistic effects of key process parameters; proposing a dual-dimensional coating classification system based on composition and function; and constructing a quantitative database encompassing 11 key performance indicators. This work provides theoretical foundations and data-driven guidance for the precise design and selection of high-performance HEBM coatings.

Keywords: high-energy ball milling (HEBM); coating element screening; process parameter synergy; coating classification

1. Introduction

Coating technology, as an integral component of modern industry and advanced manufacturing, is projected to achieve a global market value exceeding \$250 billion by 2030. Its performance is a critical determinant in the service life of aerospace engines, the safety of energy storage systems, and the success rate of medical implants [1]. Conventional coating techniques, such as chemical vapor deposition (CVD) and electrochemical deposition, though widely used, are often hampered by challenges including high energy consumption, complex processes, and limitations in achievable compositions [2]. These limitations restrict their capability to meet the growing demand for multifunctional coatings capable of operating in extreme environments.

In this context, high-energy ball milling (HEBM), which achieves atomic-level alloying and the synthesis of nanocrystalline or amorphous materials through intense mechanochemical activation, presents a compelling alternative for coating preparation. The motivation for employing HEBM stems from its several distinct advantages over conventional vapor-phase and liquid-phase deposition methods. First, from an economic and environmental perspective, HEBM is a predominantly solid-state process that can reduce energy consumption by 60%–80% compared to high-temperature CVD processes [3]. Second, it offers unparalleled compositional flexibility, enabling the synthesis of novel coating materials that are difficult or impossible to obtain by other means, such as complex high-entropy alloys [4]. Third, This technique enables the simultaneous tuning of multiple coating properties, including hardness, wear resistance, and functional characteristics, through parameter control [5]. Finally,



Academic Editor: Raul Arrabal

Received: 12 September 2025

Revised: 15 October 2025

Accepted: 21 October 2025

Published: 19 November 2025

Citation: Qi, Z.; Zhang, H.; Guo, X.; Geng, L. Preparation and Classification of Coatings by High-Energy Ball Milling: A Review. *Coatings* **2025**, *15*, 1343. <https://doi.org/10.3390/coatings15111343>

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HEBM can facilitate excellent interfacial bonding with substrates through mechanisms like mechanical interlocking and interdiffusion, which is crucial for coating durability.

Previous research has laid a foundation for understanding HEBM's potential in coating fabrication. Studies have demonstrated its efficacy in producing various coating systems, from conventional metallic alloys to advanced ceramics [6]. However, existing literature often focuses on specific material systems or isolated process-property relationships. A significant gap remains in the systematic integration of elemental screening, process parameter synergy, and a coherent classification framework for HEBM coatings. This fragmentation hinders the development of predictive models and rational design principles.

To address the aforementioned challenges, this study proposes a synergistic research framework integrating “elemental screening, parameter optimization, and functional design” to systematically review the preparation mechanisms and performance regulation principles of HEBM coatings. The innovations are reflected in (1) establishing a two-tier screening criterion for HEBM elements, constructing a usage framework for 61 elements, and further providing elemental recommendations based on the functional requirements of target coatings; (2) revealing the influence patterns of milling parameters on coating properties; (3) constructing a dual-dimensional classification system based on composition and function, thereby linking coating selection with process parameters.

2. Criteria for Coating Element Screening

Not all 118 natural elements are suitable for HEBM coating preparation. Fourteen transuranic elements, being synthetic with extremely short half-lives, generally exist only under laboratory conditions and are exceptionally scarce, thus being excluded from coating applications. Based on melting and boiling points, gaseous elements (H, He, N, O, F, Ne, Cl, Ar, Kr, Xe, Rn) and liquid elements (Br, Hg) exhibit very low melting and boiling points, existing in gaseous or liquid states under most conditions. However, coating applications require solid coatings, hence these are also excluded.

Actinides (Ac, Th, Pa, U, Np, Pu, Pm, Am, Cm, Bk, Cf, Es, Fm, Md, No, Lr) are radioactive, these elements spontaneously emit particles or radiation from unstable nuclei, posing risks of radioactive contamination and severe health hazards, thus rendering them unsuitable for coating applications. Additionally, technetium, polonium, astatine, francium, and radium are excluded due to their radioactive nature [7].

Elements such as As, Sb, Cd, Hg, Pb, Be, Tl demonstrate extreme toxicity. Even trace exposure can induce neurological damage, hepatorenal dysfunction, immune system impairment, and carcinogenesis, disqualifying them from use in coatings intended for industrial or consumer applications.

Alkali metals (Li, Na, K, Rb, Cs) are excluded due to their hyperreactivity with oxygen and water under ambient conditions. This instability compromises coating durability and introduces safety risks.

The halogen group (F, Cl, Br, I, At) exhibits strong chemical reactivity. Their polymeric or compound forms may generate coatings with latent health and environmental hazards, warranting exclusion from material selection [8].

Suitable elements include alkaline earth metals (Be, Mg, Ca, Sr, Ba), post-transition metals (Al, Ga, In, Sn, Bi), metalloids (B, Si, Ge, Te), non-metals (C, N, O, P, S, Se), transition metals (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Zr, Nb, Mo, Ru, Rh, Pd, Ag, Hf, Ta, W, Re, Os, Ir, Pt, Au), and rare earth elements (Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu), totaling 61 elements.

Alkaline Earth Metals exhibit relatively low Vickers hardness values. Calcium, strontium, and barium show hardness below 100 HV, while pure magnesium ranges between 30–40 HV. Due to their high chemical reactivity, these metals are typically alloyed for

coating applications. Qiu et al. [9] developed an eco-friendly hybrid coating on AZ31 magnesium alloy substrates, significantly improving corrosion resistance.

Post-Transition Metals demonstrate varied hardness profiles: gallium and thallium range between 10–20 HV, indium at 15–25 HV, lead at 50–70 HV, aluminum at 30–60 HV, and tin at 70–90 HV. Aluminum, in particular, is widely utilized in industrial applications owing to its lightweight nature and the corrosion-resistant properties of its oxide Al_2O_3 . Xue et al. [10] synthesized Al_2O_3 -Ru composite coatings doped with noble metals, achieving minimal oxidation rate (K) and oxide fragmentation (G).

Metalloids possess semi-metallic characteristics, making them suitable for semiconductor and integrated circuit coatings. Ma et al. [11] optimized Al-Si coatings with varying silicon content, demonstrating peak oxidation resistance at 8 wt% Si.

Non-Metals, though incapable of forming standalone coatings, play critical roles in coating synthesis. For instance, carbon doping enhances metal alloy hardness, while oxygen facilitates oxide layer formation. Zhou et al. [12] maximized TiAlN coating 31.60 GPa hardness by adjusting carbon content to 14.76%, achieving Ti-N to Ti-C bond saturation. Zhao et al. [13] attributed the conductivity of TiN_x coatings to three factors: metallization of Ti 3d orbitals, reduced N 2p orbital contributions, and lattice contraction from nitrogen vacancies.

Transition Metals exhibit the most diverse properties in the periodic table. Tungsten displays extreme approximately 3430 HV hardness, while pure copper and iron range between 100–150 HV and 160–220 HV, respectively. Gözl et al. [14] fabricated Fe-Cr-Mo coatings with Mo_2FeB_2 , achieving microhardness of 754.67 ± 28.80 – 1811 ± 25 HV_{0.3}. Shubham et al. [15] reported a high-entropy FeCoNiMnCu alloy coating with 325 HV hardness, underscoring the efficacy of alloy-based coatings.

Rare Earth Elements exhibit comparable 160–240 HV hardness and enable multifunctional coatings with enhanced corrosion resistance, magnetism, and optical properties. Singh et al. [16] fabricated a novel Al_2O_3 - La_2O_3 composite coating using the rare earth element La, achieving a hardness of up to 3.38 GPa.

Following the initial feasibility screening for environmentally compatible elements, a second-tier functional screening can be conducted to optimize the selection from the 61 candidate elements based on the functional requirements of the target coating. This process correlates the intended coating performance with the inherent physicochemical properties of the elements, enabling performance-oriented design.

To achieve high hardness and wear resistance, priority should be given to elements that form strong covalent-bonded ceramic phases, such as B, C, N, and Si, which are essential for synthesizing ultra-hard phases like TiB_2 , SiC, and TiN [17]. Simultaneously, refractory metals with high melting points and high modulus, such as W, Mo, Ta, and Re, can significantly enhance the strength and high-temperature stability of metal-based coatings through solid-solution strengthening [18].

To ensure toughness and bonding strength—thereby avoiding brittle fracture or delamination of the coating—metals with good plasticity and toughness should be incorporated as binder phases or matrices. Face-centered cubic metals such as Co, Ni, and Cu are ideal choices [19], as they effectively blunt crack propagation and enhance interfacial adhesion between the coating and the substrate.

For applications in high-temperature or corrosive environments, elements such as Al, Cr, and Si are preferred due to their ability to form dense protective films like Al_2O_3 , Cr_2O_3 , or SiO_2 [20]. Furthermore, the addition of trace amounts of rare earth elements such as Y, La, and Ce can utilize their reactive nature to refine the grains of the oxide scale, improve its adhesion, and prevent spallation.

Specific elemental characteristics can also impart specialized functional properties to coatings. For instance, Ag and Cu may be introduced for antibacterial purposes [21], and Ag, Cu, Au, and carbon in graphene structures are preferred for high electrical or thermal conductivity, while semiconductor elements such as Si and Ge are crucial for semiconductor or optoelectronic applications [22].

Toxicity Considerations: Improper elemental selection severely compromises the biocompatibility and environmental sustainability of HEBM coatings. Global toxicity incidents in 2010–2022 reveal that 78% were linked to heavy metal ions like Cr^{6+} and Cd^{2+} [23]. Thus, systematic elemental screening is critical. The established criteria identify 61 non-radioactive/low-toxicity elements as viable candidates for HEBM coatings. These elements are mapped into three design frameworks:

Metallic Coatings: Utilize transition metals and rare earth elements, leveraging solid-solution strengthening or high-entropy effects.

Ceramic Coatings: Exploit strong covalent bonds of metalloids/non-metals to form oxides, carbides, or nitrides.

Composite Coatings: Achieve functional gradient designs through metal–ceramic combinations or multi-element doping.

3. HEBM Coating Preparation Mechanisms

The fundamental principle of HEBM relies on mechanical energy to induce physical fragmentation and chemical bonding within materials. Three classical theories elucidate its operational mechanisms: the Pure Two-Phase Motion Theory, the Three-Phase Mixed Motion Theory, and the Reniform Creep Theory.

(1) Pure Two-Phase Motion Theory

This theory asserts that the milling process alternates between two dominant phases. **Impact Phase:** Characterized by high-energy collisions resulting from the free fall of grinding balls, which fracture large particles and propagate cracks. **Friction Phase:** Defined by shear stress generated through sliding friction between grinding balls and the container wall, refining particles and promoting atomic diffusion [24].

(2) Three-Phase Mixed Motion Theory

Expanding upon the two-phase model, this theory introduces a Random Collision Phase, proposing that milling dynamics arise from the combined effects of three motion modes: impact, friction, and stochastic collisions [25]. Key mechanisms include: **Localized Extreme Conditions:** Transient temperatures ranging from 200 °C to 800 °C facilitate solid-state reactions, while pressures of 1–5 GPa enable phase transitions such as graphite-to-diamond conversion. These conditions also explain the formation of amorphous and metastable phases in metal alloys.

(3) Reniform Creep Theory

Reniform Creep Theory describes the interaction dynamics between grinding media and materials within ball mills, proposing that grinding balls follow periodic kidney-shaped creeping trajectories inside the milling chamber [26]. This motion mechanism achieves material plastic deformation and homogeneous mixing through quasi-static compression-relaxation cycles characterized by low-frequency, high-amplitude compressive and release actions [17]. Grinding balls densely pack under static pressures of 10–100 MPa, fracturing particles and displacing grain boundaries. Shear friction between balls refines particles, while viscoelastic recovery suppresses cold welding phenomena. Table 1 provides a comparative overview of the three theories, including their dominant forces, applicable materials, energy efficiency, and industrial applications. Energy efficiency refers to the

percentage of the total input mechanical energy that is utilized to induce powder particle fracture, alloying, or chemical reactions, with the remaining energy predominantly dissipated as heat.

Table 1. Theoretical comparison and selection guide.

Theory Type	Dominant Forces	Applicable Materials	Energy Efficiency	Industrial Applications	Documentary Basis
Pure Two-Phase Motion	Impact + Shear	Brittle ceramics/intermetallics	35%–50%	Nano-ceramic powder preparation	[27]
Three-Phase Mixed Motion	Impact + Shear + Random Collision	Metals/alloys	45%–60%	High-entropy alloy amorphization, nitride coatings	[28]
Reniform Creep	Quasi-static compression + Viscoelastic dissipation	Polymers/composites	25%–40%	Graphene dispersion, drug nanoparticles	[29]

Energy efficiency: the percentage of the total input mechanical energy utilized for processes including particle fracture, alloying, or chemical reactions.

In summary, the three classical theories of HEBM correspond to distinct dominant mechanisms and applicable scenarios:

The Pure Two-Phase Motion Theory is suitable for processing brittle ceramics or intermetallic compounds, where its high-energy impact and shear mechanisms effectively achieve material fracture and nanonization [27].

The Three-Phase Mixed Motion Theory is more appropriate for describing the ball milling process of metals and alloys. The stochastic collision phase inherent in this theory helps explain complex kinetic behaviors such as amorphization, mechanical alloying, and the synthesis of nitride coatings [28].

The Reniform Creep Theory demonstrates advantages in wet milling or when handling polymers, composites, or high-viscosity systems. Its quasi-static compression–relaxation cycles facilitate uniform material mixing and plastic deformation while effectively suppressing cold welding [29].

4. HEBM Coating Process System

The core process chain for fabricating coatings through HEBM can be deconstructed into three critical stages: raw material formulation and loading, mechanical alloying (MA), and post-processing with coating deposition. The technological essence lies in achieving powder nanonization and interfacial metallurgical bonding through high-energy solid-state reactions. The detailed workflow is illustrated in Figure 1 [30].

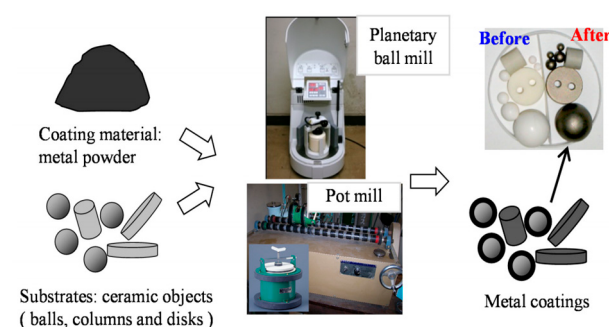


Figure 1. The process of preparing the coating by high-energy ball milling [30].

The process flow depicted in Figure 1 comprises three stages: raw material formulation and loading optimization, the kinetics of mechanical alloying, and post-processing with coating deposition. These stages are described below.

(1) Raw Material Formulation and Loading Optimization

Based on the compositional design of the target coating, metallic or ceramic powders are loaded into the milling container at a predetermined ball-to-powder ratio ranging from 5:1 to 100:1. The grinding media used in ball mills, typically highly hard materials such as WC or ZrO₂ balls [31], are selected for their stable chemical properties, which minimize chemical reactions with the raw materials. Nevertheless, after determining the raw materials, the grinding balls must also be carefully selected to avoid any impurity formation resulting from chemical reactions during HEBM. For raw materials with high hardness, the grinding media should comply with the hardness matching criterion to prevent the generation of wear debris from the balls. Furthermore, the introduction of an inert atmosphere helps suppress chemical reactions, thereby preserving the chemical purity of the powders. Strict selection of grinding balls not only ensures the quality of the HEBM process but also reduces contamination and wear of both the milling container and the grinding media [32].

(2) Kinetics of Mechanical Alloying

During high-speed operation of the ball mill, the kinetic energy of grinding balls induces cyclic cold welding and fracture behaviors in the powder particles. This process involves plastic deformation, phase transformation control, and nanostructural evolution. Localized temperature increases ranging from 200 °C to 500 °C promote grain boundary migration and elemental interdiffusion [33], while grain refinement to 10–50 nm significantly enhances the specific surface area of the material [34].

(3) Post-Processing and Coating Deposition

The milled products undergo heat treatments such as annealing or tempering to stabilize phase structures and alleviate residual stresses. Under specific conditions, substrates introduced into the HEBM process can directly form coatings. Subsequent deposition techniques, including thermal spraying or laser cladding, deposit nanocomposite powders onto substrates, enhancing the coating's interfacial bonding strength [35].

Compared to conventional coating technologies like magnetron sputtering and vapor deposition, HEBM offers superior compositional flexibility and energy efficiency. For instance, it enables the synthesis of complex coatings containing rare-earth elements or high-entropy alloys, with energy consumption per unit mass approximately one-fifth of that required for magnetron sputtering [36].

However, HEBM necessitates strict exclusion of radioactive elements and highly toxic substances through the elemental screening criteria outlined in Section 2.

4.1. Selection and Working Mechanism of HEBM Equipment

HEBM technology can be categorized into three mainstream equipment types based on energy input mechanisms: planetary ball mills, vibratory ball mills, and stirred ball mills [37]. These systems differ fundamentally in their mechanical energy transfer efficiency to powders and their applicability across various scenarios.

The planetary ball mill operates on the principle of combined planetary and rotational motion, generating high centrifugal acceleration. The multidirectional motion produces shear forces that improve grinding uniformity, while its high energy density enables highly homogeneous milling [38]. Due to its high energy density and excellent process controllability, the planetary ball mill dominates laboratory-scale coating material development, particularly in the preparation of high-performance nanocrystalline ceramics and alloy coatings. Its limitations include reduced loading efficiency resulting from the multi-container design and the need for external cooling systems to suppress temperature rise.

The vibratory ball mill utilizes high-frequency vibrations to drive grinding media for material pulverization and mixing. The intense vibrational energy input enables rapid

and effective particle size reduction, achieving ultrafine powders [39]. Vibratory ball mills excel in the rapid alloying of metal-based coatings and are suitable for metal powders and alloys. Compared to other rotary ball mills, they feature lower operational noise and simpler handling. However, they struggle to process large or extremely hard materials directly and often require pre-crushing steps.

The stirred ball mill employs impeller-generated laminar shear forces for material fragmentation and homogenization. Its advantage lies in its particular suitability for processing viscous or high-viscosity materials [40], making it irreplaceable for handling composite coating materials that are sensitive to shear forces or require preservation of specific morphologies. The stirring action prevents material adhesion to container walls and simplifies operation. However, the requirement for high rotational speeds increases noise levels and accelerates equipment wear when excessively high speeds are used.

Among these methods, planetary and vibratory ball mills are more widely adopted for high-energy milling due to their low noise, high energy output, and cost-effectiveness. Planetary ball mills, in particular, dominate laboratory-scale applications owing to their versatility and precision.

4.2. Regulation of HEBM Process Parameters

The properties of ball-milled products are highly sensitive to parameter adjustments, with different milling parameters critically influencing the final outcomes [41]. As the variety of coatings prepared via high-energy ball milling continues to expand, researchers have observed that identical parameters may yield divergent effects across materials under similar conditions. Key parameters governing coating performance include dry/wet milling, grinding ball diameter, milling atmosphere, ball-to-powder ratio, rotational speed, and milling time.

(1) Dry/Wet Milling

Dry milling and wet milling represent two distinct methodologies in the ball milling process, each optimized for specific application scenarios. Dry milling operates without liquid media, minimizing unintended chemical reactions—particularly advantageous for materials reactive with water or solvents—while simplifying post-processing by eliminating liquid handling. In contrast, wet milling produces finer and more uniformly distributed particles with reduced agglomeration, aided by the cooling effect of liquid media. Bankowska et al. [42] demonstrated enhanced reactivity and solubility of limestone through dry milling, while Yao et al. [43] achieved reduced surface roughness in $\text{Al}_2\text{O}_3/\text{TiO}_2$ composite coatings via wet chemical-mechanical grinding.

(2) Grinding Ball Diameter

The diameter of grinding balls constitutes a critical parameter in HEBM processes. Ball diameter directly impacts particle size and milling efficiency due to collision dynamics between grinding media and materials. Typical diameters range from 3–20 mm, with materials including hard alloys, stainless steel, and ceramics. Hu et al. [44] elucidated energy transfer mechanisms in planetary ball mills, confirming the direct correlation between ball diameter and product particle size. Celep et al. [45] observed coarser products with larger balls, while Yao Guangqian [46] reported reduced grinding efficiency at excessive diameters due to cascading motion, lower average acceleration, and diminished collision frequency.

(3) Milling Atmosphere

The gaseous environment within the mill crucially regulates chemical reactions, oxidation/reduction processes, and product quality. Jin et al. [47] achieved 90.04% Ti-enriched material purity using CO_2 atmosphere milling combined with magnetic separation. Under pressurized N_2 , Salah et al. [48] reduced SnSe_2 polycrystals to nanoscale while suppressing

thermal conductivity and enhancing thermoelectric performance. Luo et al. [49] synthesized nanostructured $\text{ZrB}_2\text{-TiB}_2$ under argon protection.

(4) Ball-to-Powder Ratio (BPR)

The ball-to-powder ratio is defined as the mass ratio of grinding balls to raw material. A higher ball-to-powder ratio accelerates the alloying process of the raw material. This occurs because an increased volumetric fraction of grinding balls enlarges the contact area between the balls and the raw material, thereby raising the collision frequency. As a result, more energy is transferred from the high-speed grinding balls to the composite material per unit time. Furthermore, the ball-to-powder ratio significantly influences the specific surface area, particle size, morphology, and grain size of the raw material in HEBM. Typical ball-to-powder ratios for HEBM range from 5:1 to 100:1. Yu Haoran et al. [50] found that higher ball-to-powder ratios widen the sintering temperature window but lead to more pronounced shrinkage before and after sintering, effectively enhancing the hardness and performance stability of the material after heat treatment. Salleh et al. [51] analyzed the effects of HEBM parameters and their interactions on alloy performance using a 16-run two-level fractional factorial design. They conducted variance analysis, regression analysis, and R^2 testing, with Figure 2 illustrating the effects of varying milling parameters on the elastic modulus (a) and mass loss (b) of the alloy. Sivasankaran et al. [52] observed substantial improvements in the ultimate strength and hardness of the alloy as the ball-to-powder ratio increased.

In the figure, the hard tissue is primarily influenced by compressive stress; thus, the elastic modulus of magnesium-zinc alloy serves as the evaluation metric. The elastic modulus of magnesium slightly increases with zinc addition. Since the modulus of solute zinc atoms exceeds that of pure magnesium, zinc incorporation predominantly enhances the modulus of magnesium.

(5) Rotational Speed of Ball Mill (RPM)

The motion patterns of grinding balls in the ball mill determine milling efficiency. When the mill structure, ball-to-powder ratio, and process control agents remain constant, the motion of grinding balls depends entirely on rotational speed. Generally, higher rotational speeds yield smaller material particle sizes. Zhang Fuxian et al. [53] observed particle size reductions of 36.2%, 46.7%, and 58.1% at rotational speeds of 400, 600, and 800 r/min, respectively. Hussain Imad et al. [54] reported increased magnetization in alloys at higher speeds, attributing this to reduced surface-area-to-volume ratios that enhance ferromagnetic interactions. Chen Jiandong et al. [55] demonstrated optimized milling efficiency by adjusting rotational speeds at different stages of intermittent wet ball milling.

(6) Milling Time (MT)

Prolonged milling time in HEBM increases cumulative energy transfer to materials through sustained compression and grinding. This facilitates thorough physical and chemical reactions under elevated temperatures. Precise control of milling duration is critical for achieving desired product characteristics. Wang et al. [56] documented progressive particle refinement with extended milling time, albeit accompanied by increased impurity content. Wei et al. [57] observed significant enhancements in room-temperature Vickers hardness and compressive strength due to grain refinement, consistent with the Hall–Petch law. Qi et al. [58] investigated the effects of ball milling time on various properties of TiO_2 samples, including micromorphology and crystal structure. Figure 2 displays the Raman spectra of titanium oxide subjected to different ball milling durations.

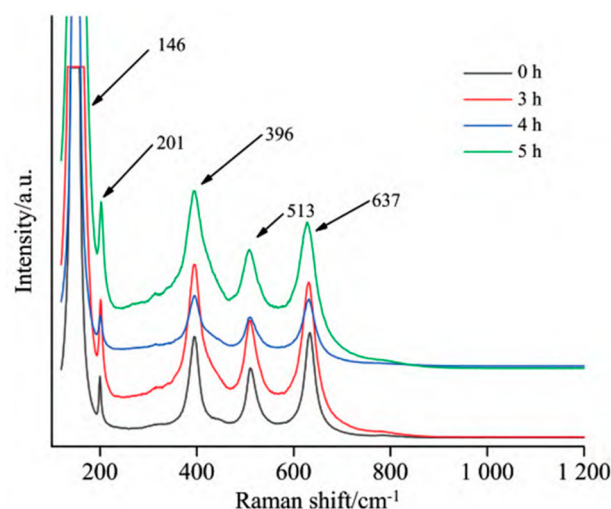


Figure 2. Raman spectrum of TiO₂ with different milling time [58].

Rotational speed and milling time universally govern energy transfer frequency and duration in HEBM, establishing them as the two most critical parameters for process control.

4.3. Multi-Parameter Synergistic Effects Response Surface Curve Analysis

The previous section provided a fundamental introduction to the parameters of HEBM. However, in the actual preparation of coatings via HEBM, the outcomes are influenced not by a single parameter, but by the synergistic effects of multiple milling parameters. To investigate the impact of these parameters on the final coating quality, this review employs an experimental study on the preparation of nano-scale Cu_xO and TiO₂ coatings via HEBM on 2 × 2 mm stainless steel substrates. A mathematical model was developed using Design Expert 10 software [59].

Based on a Box–Behnken experimental design, the ball-to-powder ratio (5:1–15:1), rotational speed (400–1000 r/min), and milling time (6–24 h) were selected as independent variables, while hardness, wear resistance, and salt spray corrosion resistance duration were used as response variables for coating performance. A quadratic regression model was constructed. Model validation identified the optimal parameter combination as a ball-to-powder ratio of 10:1, a rotational speed of 700 r/min, and a milling time of 15 h. A ball-to-powder ratio of 10:1 helps balance grain refinement and stress mitigation, a rotational speed controlled near 700 r/min ensures sufficient energy input without overheating, and a milling time of 15–24 h guarantees process efficiency while preserving equipment longevity. Under this optimal combination, the predicted coating performance metrics are: hardness of 400–410 HV, a wear resistance coefficient μ of 0.65–0.72, and a salt spray corrosion resistance time of 240–270 h, with deviations from experimental measurements below 5%. Insufficient or excessive milling can significantly affect the coating thickness. The measured coating thickness consistently exceeded 30 μ m, indicating good coating adhesion and the formation of an interface structure characterized by metallurgical bonding/mechanical interlocking. The coefficient of determination (R^2) for the model is 0.972, indicating that the model explains 97.2% of the variability in the experimental data response values, demonstrating high significance and predictive accuracy for reliably analyzing the relationship between process parameters and coating performance.

The coefficient equation for hardness is expressed as: $\text{Hardness} = 401.00 + 37.50A + 22.50B + 22.50C + 10.00AB + 10.00AC - 28.00A^2 - 8.00B^2 - 13.00C^2$.

Analysis of variance (ANOVA) revealed that the effect of the ball-to-powder ratio is highly statistically significant ($p < 0.0001$) with a contribution rate of 37.5%, indicating

its powerful and decisive influence on coating hardness, not attributable to random error. As shown in Figure 3a–c, the interaction between the ball-to-powder ratio and milling time exhibits non-linear regulatory effects on wear resistance ($\Delta\mu = \pm 0.12$) and corrosion resistance ($\Delta t = \pm 30$ h). The response surface curves illustrating the effects of the multiple parameters are presented in Figure 3a–c.

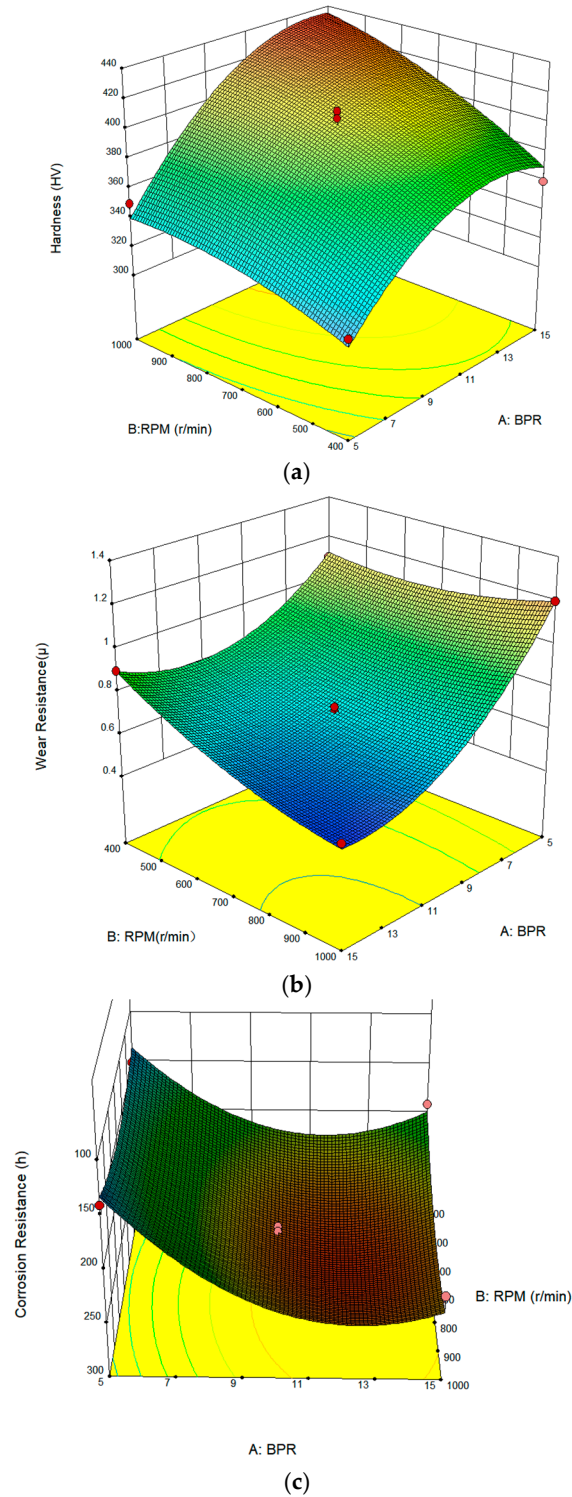


Figure 3. Response surface curves of hardness (a), wear resistance (b), and corrosion resistance (c) under the influence of multiple parameters.

This parameter combination optimizes the synergistic effects on coating performance. The ball-to-powder ratio governs alloying kinetics and grain refinement, the rotational speed modulates energy input and particle morphology, and the milling time dictates the degree of reaction completion. Together, these factors collectively enable performance enhancement [60]. In summary, while an excessively high ball-to-powder ratio may accelerate alloying, it risks inducing stress concentration—a concern mitigated by the 10:1 ratio. Similarly, unduly high rotational speeds elevate temperatures, whereas 700 r/min maintains effective energy input without thermal degradation. An appropriate milling duration ensures thorough reactions while preserving equipment service life.

4.4. Post-Processing and Coating Deposition Techniques

As researchers demand increasingly multifunctional coatings, HEBM-fabricated coatings are often combined with additional deposition techniques to achieve novel performance characteristics that are difficult to obtain by any single method. Powders prepared via HEBM can serve as advanced feedstock materials, which are subsequently deposited or consolidated using various coating techniques to form or enhance the final coating. Compatible deposition methods that can be integrated with HEBM include sol–gel processing, encapsulation, chemical vapor deposition (CVD), physical vapor deposition (PVD), laser cladding, electrochemical deposition, plasma spraying, and in situ synthesis.

(1) Sol–Gel Method

The sol–gel method involves applying inorganic or organic-inorganic composite coatings synthesized via sol–gel chemistry onto HEBM-processed powders, enabling nanostructural control. Nezamdoust et al. [61] coated magnesium alloys with sol–gel anti-corrosion layers containing hydroxylated nanodiamond particles, achieving defect-free coatings with superior corrosion resistance.

(2) Encapsulation

Encapsulation deposits protective or functional layers onto alloy powders post-HEBM through physical or chemical means, ideal for metallic or metal oxide coatings. Deng Haotian et al. [62] applied dual lithium titanate and polypyrrole encapsulation on LiCoO₂ via solid–liquid coating, enhancing surface protection and ionic/electronic conductivity.

(3) Chemical Vapor Deposition (CVD)

CVD deposits thin films on HEBM powders by thermally decomposing gaseous precursors in a reactor, producing high-quality ceramic or carbon-based coatings. Hu et al. [63] synthesized smooth, spherical SiC coatings on graphite substrates using methylsilane as a gas source.

(4) Physical Vapor Deposition (PVD)

PVD employs sputtering or evaporation in vacuum environments to deposit coatings onto HEBM powders, suitable for metals, alloys, and ceramics. Xiao et al. [64] fabricated low-surface-roughness TiAlN coatings on fine-grained Ti(C,N)-based cermets, significantly improving wear resistance.

(5) Laser Cladding

Laser cladding modifies surface microstructures or introduces new phases to HEBM-processed powders/components, enabling localized hardening. Liu Ran et al. [65] developed ZrC-reinforced CoCrNi alloy coatings on mild steel, achieving a maximum hardness of 6379.8147 MPa and a friction coefficient of 0.161.

(6) Electrochemical Deposition

This technique electrochemically deposits metallic coatings on conductive HEBM substrates. Liu Pin et al. [66] constructed bioactive hydroxyapatite (HA) coatings on porous tantalum scaffolds, accelerating osteoblast adhesion and bone-like apatite deposition.

(7) Plasma Spraying

Plasma spraying projects molten coating materials onto HEBM-treated substrates via high-velocity plasma jets, ideal for high-temperature, wear-resistant coatings. Ma Baoxia et al. [67] produced TiB₂-TiC-Co ceramic coatings on Q235 steel with excellent bond strength and sliding wear resistance.

(8) In Situ Synthesis

In situ synthesis generates protective or functional coatings through precursor-induced reactions on powder surfaces. Yin et al. [68] fabricated Fe-Al/Al₂O₃ composite coatings via rare-earth-modified aluminizing and in situ synthesis.

Synergistic combinations of methods—such as sol-gel with PVD—may concurrently enhance hardness and corrosion resistance, warranting focused research for next-generation multifunctional coatings.

5. Coating Classification System and Performance Quantitative Prediction

5.1. Coating Classification

Based on the screening of chemical elements suitable for HEBM as established in Section 2, combined with appropriate coating deposition techniques, HEBM enables the fabrication of coating systems with diverse elemental combinations. The coating classification framework proposed in this study not only considers chemical composition characteristics but, more importantly, incorporates the unique process features of HEBM and their effects on coating microstructure and performance properties. This classification system demonstrates strong compatibility with HEBM technology, as the classification criteria directly reflect the initial characteristics of powder materials, mechanical alloying pathways, and the resulting phase composition and interface structure developed during the HEBM process.

According to primary composition characteristics, HEBM coatings can be categorized into three main types: metallic coatings, ceramic coatings, and composite coatings. This classification system not only provides valuable reference for material design of HEBM coatings but also enables effective pre-assessment of process parameters and selection of compatible deposition methods. The framework establishes crucial connections between coating categories and their corresponding HEBM process optimization strategies, allowing for systematic matching of coating types with their ideal processing conditions and deposition techniques.

(1) Metallic Coatings

Metallic coatings enhance substrate performance through the deposition of metallic materials and are divided into two categories: single-metal coatings and multi-principal element alloy coatings.

Single-Metal Coatings, composed of a single metallic element (e.g., zinc, nickel, chromium), serve specialized roles: Zinc coatings are widely used in bridge structures and automotive chassis for corrosion protection, with salt spray resistance exceeding 500 h. Chromium coatings are applied to cutting tools for superior wear resistance.

Multi-Principal Element Alloy Coatings consist of alloys with multiple metallic elements, categorized into high-entropy alloy coatings and medium-entropy alloy coatings. The elements typically exist in equiatomic or near-equiatomic ratios. High-entropy alloys generally comprise 5–13 principal elements in equiatomic or near-equiatomic propor-

tions [69], while medium-entropy alloys contain 3–4 principal elements, forming materials with complex crystal structures. Multi-principal element alloy coatings exhibit enhanced high-temperature stability, oxidation resistance, and wear resistance due to their unique high-entropy characteristics. High-entropy alloy coatings demonstrate a 300% increase in hardness compared to traditional alloys [70] and are used in aerospace engine blades and nuclear power components.

As an emerging frontier in surface engineering, multi-principal element alloy coatings—particularly high-entropy alloy (HEA) coatings—are composed of multiple principal elements in near-equiatomic ratios. Their exceptional comprehensive properties stem from four core effects: the high-entropy effect, which stabilizes solid-solution phases; severe lattice distortion, which impedes dislocation movement; sluggish diffusion, which enhances thermal stability; and the “cocktail” effect, which synergistically optimizes performance. These mechanisms collectively enable HEA coatings to achieve a hardness increase of up to 300% compared to conventional alloys [71], along with excellent corrosion resistance and high-temperature stability. Consequently, such coatings show great potential for protecting critical components in extreme environments, such as those encountered in aerospace and nuclear power applications.

(2) Ceramic Coatings

Ceramic coatings are inorganic non-metallic coatings characterized by exceptional high-temperature resistance, wear resistance, corrosion resistance, and chemical stability. These coatings can be prepared via HEBM and are categorized into oxide ceramic coatings and carbide/nitride/boride ceramic coatings.

Oxide Ceramic Coatings combine high-temperature stability, oxidation resistance, and wear resistance, typically operating above 1200 °C with hardness values ranging from 1500–2200 HV [72]. Carbide/Nitride/Boride Ceramic Coatings primarily consist of hard coatings with extreme hardness and superior wear resistance. These coatings exhibit hardness values between 2000–3500 HV and wear rates as low as 0.005 mm³/N·m, making them ideal for cutting tools, molds, and wear-resistant components.

Certain ceramic coatings also exhibit specialized properties such as electrical insulation or antibacterial effects. Arifa et al. [73] demonstrated that higher-purity Al₂O₃ powders yield coatings with increased volume resistivity. Ding et al. [74] developed Ag⁺-loaded TiO₂ coatings (Ag-TiO₂) with enhanced antibacterial activity against *Staphylococcus aureus*, proportional to Ag⁺ concentration.

(3) Composite Coatings

Composite coatings refer to coatings composed of two or more distinct materials, which can include metals, ceramics, polymers, or other inorganic non-metallic materials. These coatings combine the advantages of multiple materials to deliver superior comprehensive properties such as biocompatibility, wear resistance, corrosion resistance, thermal stability, and mechanical strength. The performance benefits of composite coatings arise from their multi-scale structural design. While classification by composition can be overly complex due to the diversity of materials, composite coatings can be categorized into three types based on the dual dimensions of reinforcement distribution patterns and interfacial bonding mechanisms: particle-reinforced composite coatings, reaction-synthesized composite coatings, and layered composite coatings.

Particle-reinforced composite coatings such as Al-Al₂O₃-ZrO₂ are applicable in aerospace applications. Cai et al. [75] developed Al-Al₂O₃-ZrO₂ composite refractory materials exhibiting exceptional oxidation resistance, erosion resistance, and thermal shock stability. Reaction-synthesized composite coatings like TiB₂-TiC-Co were fabricated by Ma et al. [67] on Q235 steel substrates. Layered composite coatings such as Ti/Al₂O₃

multilayer coatings demonstrate a threefold lifespan extension compared to homogeneous Al_2O_3 coatings. Dercz et al. [76] prepared Ti/ ZrO_2 and Ti/ Al_2O_3 composite coatings via powder metallurgy.

The article provides a detailed classification of coating compositions and introduces a dual-dimensional taxonomy for complex composite coatings as illustrated by the coating classification dendrogram in Figure 4. Based on this classification system, quantitative prediction of coating performance requires integration with the process parameter synergy mechanisms discussed in Section 3.

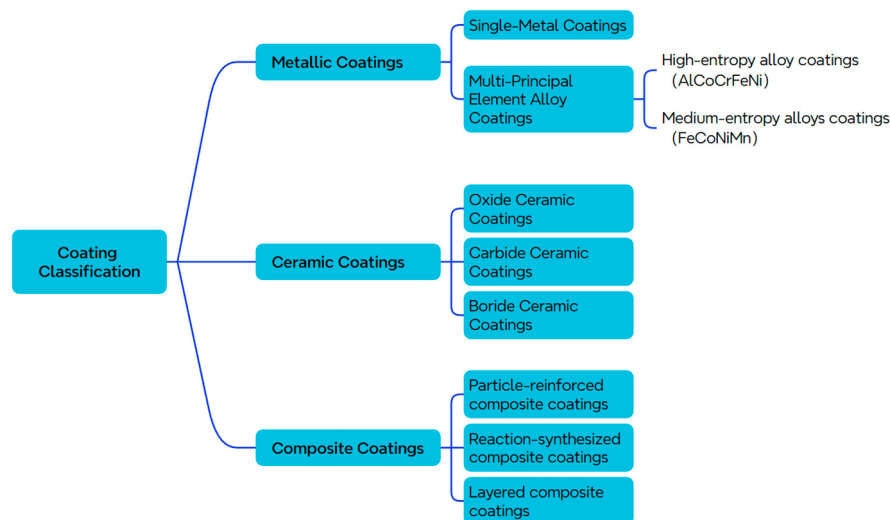


Figure 4. Taxonomy Logic Diagram of Coatings.

For metallic coatings, the ball-to-powder ratio can be controlled between 5:1 and 30:1, with rotational speed adjusted according to metal hardness and milling time regulated to influence alloying degree. This achieves solid-solution strengthening effects in high-entropy alloy coatings as demonstrated in Figure 3a.

For ceramic coatings, a rotational speed of 600–1000 r/min combined with dry/wet milling selection from Table 1 reduces grain boundary defect density. Taking Al_2O_3 as an example, this approach enables salt spray corrosion resistance exceeding 4000 h as shown in Table 2.

For composite coatings, post-processing techniques such as laser cladding synergize with milling parameters. For instance, TiB_2 -TiC coatings exhibit gradient-distributed reinforcement phases, as depicted in Table 2, optimizing wear resistance to $\mu = 0.15$.

5.2. Quantitative Prediction of Coating Performance

Quantitative prediction of coating performance is achieved through a “composition-function-property” relational database, as summarized in Table 2, which integrates 96 experimental datasets across 11 metrics including hardness, wear resistance, and corrosion resistance. For metallic coatings, high-entropy alloys exhibit significantly higher hardness than traditional chromium-plated layers, a disparity attributed to the enhanced solid-solution strengthening effects of optimized ball-to-powder ratios, as detailed in Section 3. In ceramic coatings, Al_2O_3 demonstrates salt spray resistance exceeding 4000 h—a 25% improvement over magnetron-sputtered counterparts—3200 h—owing to wet milling’s suppression of grain boundary oxidation. For composite coatings, the wear rate of TiB_2 -TiC-Co inversely correlates with laser cladding temperature gradients, validating the synergistic mechanisms of post-processing parameter coordination.

Table 2. Diagram of coating types, coating parameters and application fields.

Coating Classification	Hardness (HV)	Wear Rate (mm ³ /N·m)	Coefficient of Friction (μ)	Salt Spray Resistance Time (h)	Bond Strength (MPa)	Highest Temperature Resistance (°C)	Rate of Oxidation (mm/year)	Thermal Conductivity (W/m·K)	Electrical Conductivity (S/m)	Biocompatible (Cell Proliferation Rate)	Thermal Shock Resistance (Number of Cycles)	Environmental Compatibility (ppm for Toxic Elements)	Documentary Basis
Metallic Coatings													
Single-Metal Coatings	70–100	0.6	0.6	500	50–80	200	0.5	110	1.2×10^7	Not applicable	20	<1	[77]
Multi-Principal Element Alloy Coatings	400–1000	0.01–0.05	0.20	3000	300–500	800	<0.05	15–25	1.5×10^6	Not applicable	100	<1	[38,70,71]
Ceramic Coatings													
Al ₂ O ₃ Coatings	1500–2200	0.005–0.01	0.35	4000	200–300	1200	<0.01	5–8	Non-conductive	Not applicable	200	<1	[45,67,68,76]
TiN Coatings	2000–2500	0.008	0.25	3000	400–600	600	<0.02	20–30	Non-conductive	Not applicable	150	<1	[13,17]
SiC Coatings	2500–3000	0.003	0.18	3500	500–700	1000	<0.005	120–150	Non-conductive	Not applicable	180	<1	[63]
Composite Coatings													
Al–Al ₂ O ₃ –ZrO ₂ Coatings	600–1000	0.003	0.40	2500	150–250	600	<0.1	10–15	Non-conductive	Not applicable	80	<1	[75]
TiB ₂ –TiC–Co Coatings	1200–2200	0.001–0.005	0.15	2800	600–800	1000	<0.01	25–35	Non-conductive	Not applicable	200	<1	[67]
HA/Ti Coatings	800–1200	0.02–0.05	0.45	2000	100–200	500	<0.10	5–10	5×10^{-6}	50% increase	30	<1	[78]

Note: 1. Hardness, wear rate, and salt spray resistance duration exhibit deviations between measured and predicted values within ±5%; 2. Oxidation rate and thermal conductivity errors remain below 10% compared to experimental averages from literature; 3. The detection limit for toxic elements is 1 ppm.

6. Discussion and Future Perspectives

This study systematically analyzes the process systems and performance regulation mechanisms of multifunctional coatings prepared via HEBM. The main contributions include establishing a comprehensive technical framework, advancing green manufacturing principles, and providing quantitative guidelines for coating selection.

Although HEBM demonstrates significant potential for fabricating multifunctional coatings, this review reveals substantial challenges and knowledge gaps in the field. Current research primarily focuses on process–performance relationships at the laboratory scale, while systematic investigations of parameter interactions during scale-up and associated cost control remain insufficient. At the mechanistic level, understanding of fundamental formation mechanisms—including non-equilibrium phase transitions and interfacial metallurgical bonding kinetics—requires further development to enable accurate performance prediction and regulation. Furthermore, while this study proposes green elemental screening criteria, fully integrating environmental sustainability assessments into coating design represents an ongoing challenge. Future research must transition from iterative trial-and-error approaches to a combined data-driven and mechanistic analysis paradigm to bridge the gap between laboratory research and industrial applications.

Future research directions should focus on:

- (1) **AI-Assisted Coating Design:** Constructing a ternary knowledge graph integrating material element libraries, process parameter databases, and performance databases. Graph neural networks could mine implicit composition–process–performance relationships, potentially reducing experimental sample requirements by 50%.
- (2) **Quantitative Analysis of Cross-Scale Mechanisms:** Developing a research paradigm combining multi-physics simulation with in situ characterization. Establishing a mechanism-driven digital twin system would enable accurate prediction and active control of coating performance.

Funding: This research was funded by National Natural Science Foundation of China grant number 52072055.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: No new data were created or analyzed in this study.

Conflicts of Interest: The authors declare no conflict of interest.

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