

Influence of thermodynamic properties of metallic materials on the efficiency of hydro-vacuum atomization and hydriding processes

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Abstract: *In this study, we present the key results of computer modeling and experimental series of a combined technological process that integrates hydro-vacuum atomization and hydrogenation of metallic melts. The simulations considered several representative cases in which lead, aluminum, magnesium, Armco iron, and magnesium–aluminum alloys were used as initial materials. A comparative analysis of the obtained data revealed that, during hydro-vacuum atomization, the cooling, solidification, and hydrogenation kinetics of atomized droplets are strongly governed by the specific thermodynamic characteristics of the metals, such as the solidification interval, latent heat of fusion/solidification, and specific heat capacity. It was established that the relationship between the melt overheating temperature and the width of its solidification interval constitutes a key controlling factor in the combined processes of atomization and hydrogenation. This factor is critical because it determines both the duration and the nature of the residence of atomized metallic droplets in the transitional liquid–crystalline state, thereby defining the temporal window and the depth of highly effective physico-technological influences that promote hydrogen uptake. These influences arise during the entrainment and breakup of the melt jet in the water stream and include hydromechanical activation, structural metastabilization, and interstitial hydrogen incorporation released from collapsing cavitation cavities in water due to the thermobaric energy generated during their directed cumulative implosion.*

KEYWORDS: HYDRO-VACUUM ATOMIZATION, CRYSTALLIZATION KINETICS, HYDROGENATION, STRUCTURAL METASTABILIZATION, OVERHEATING EFFECT.

1. Introduction

The use of metallic powders as solid-state hydrogen carriers opens new prospects for the development of safe, energy-efficient, and compact systems for the storage and transportation of renewable energy.

In contrast to conventional methods of hydrogen storage in gaseous or cryogenically liquefied form, metallic powder materials enable hydrogen retention in a chemically bound state (as metal hydrides formed through chemisorption mechanisms) or physically, through adsorption on the developed surface of the particles. This approach provides a substantially higher level of operational safety and allows long- term hydrogen storage under ambient conditions without the need to maintain extremely high pressures or ultralow temperatures. Moreover, the use of powder-based metal hydride materials completely eliminates the risks and operational costs associated with high- pressure composite vessels (600–700 bar) or cryogenic cooling systems operating at 20–70 K, both of which require complex infrastructure and expensive reinforced containment shells [1]. Unlike conventional hydrogen storage technologies, the retention of hydrogen in a chemically bound metal- hydride state, followed by its controlled thermal release upon heating the metal to approximately 100–300 °C, is characterized by a high degree of controllability and operational reliability. The desorption process proceeds smoothly, and its kinetics can be effectively regulated by adjusting the temperature of the hydrogenated material. This enables precise modulation of the hydrogen release rate in accordance with the specific demands of the system [2].

An equally important operational criterion is the high reversibility and stability of the hydrogenation–dehydrogenation cycles. The ability of a material to withstand numerous working cycles without significant structural degradation or loss of reversible hydrogen capacity determines the long- term durability of powder- based metal hydride storage systems and their technological applicability in the practical energy sector.

High- cycle metal hydride systems are particularly in demand in hydrogen- powered transportation, from unmanned aerial vehicles to heavy commercial machinery, as well as in autonomous and backup energy complexes for remote facilities, in decentralized

storage systems for renewable energy, and in stationary hydrogen reservoirs used in “green” metallurgy and the chemical industry [3, 4].

The successful integration of metal hydride systems into the aforementioned high- technology sectors is largely determined by the morphological and thermodynamic characteristics of the hydrogen- storage materials. A highly developed specific surface area in powder- based materials provides a dense population of active sorption sites, significantly accelerating the kinetics of diffusive or chemisorptive hydrogen uptake and saturation. However, morphological optimization alone is insufficient to overcome the fundamental limitations that hinder large- scale commercial deployment of such storage media. The primary barrier remains the stringent thermodynamic constraints associated with the high thermal stability of certain hydride phases, which result in sluggish desorption kinetics and necessitate elevated temperatures for reversible hydrogen release [5]. Moreover, these kinetic limitations become progressively aggravated over time due to the advancing structural degradation of the material. During high- temperature operation, cyclic phase hardening induces fragmentation (cracking) of the particles, followed by thermocyclic sintering and passivation [6, 7]. These processes lead to an irreversible reduction in sorption capacity and effectively negate the initial advantages associated with a highly developed specific surface area. Consequently, this indicates that the operational efficiency of reversible metal hydride systems is governed by a fundamental compromise between the thermodynamic stability of the hydride phases formed and the kinetic parameters of the sorption–desorption processes. For example, on the one hand, hydrides of light metals and their complex compounds (MgH₂, LiBH₄, Mg(BH₄)₂, AlH₃, TiH₂) exhibit exceptionally high theoretical hydrogen capacities, ranging from 4 to 18.5 wt.% H₂. However, such high storage capacities are achieved at the expense of thermodynamically robust metal–hydrogen bonds, which require substantial energy input for subsequent dissociation and shift the desorption temperature range into the domain of elevated temperatures (>250–500 °C). This is undesirable both from the standpoint of process efficiency and due to the reduction in the operational lifetime of the storage system. On the other hand, intermetallic compounds, such as Laves phases based on AB₂- and

AB₅- type alloys (LaNi₅, TiFe, ZrMn₂), enable operation under moderate, near- ambient thermal conditions (≈ 50 – 100 °C) [8, 9]. Nevertheless, their hydrogen storage capacity is significantly lower than that of light- metal hydrides and typically does not exceed 1.4–2 wt.% H₂. This limitation restricts their applicability in technical systems where high specific energy density is a critically important requirement.

A compromise-oriented attempt to resolve the thermodynamic–kinetic contradiction involves transitioning to lighter binary systems, among which intermetallic alloys of the Mg–Al system are of particular interest. The Mg₁₇Al₁₂ and Mg₂Al₃ phases occupy an intermediate position between high- capacity yet thermodynamically stable light- metal hydrides and low- temperature but low- capacity intermetallic compounds. The incorporation of aluminum into the magnesium matrix partially reduces its thermodynamic stability while maintaining a relatively high specific hydrogen capacity. For the Mg₁₇Al₁₂ phase, the theoretical hydrogen storage capacity is approximately 4.0–4.4 wt.% H₂, and due to the destabilizing influence of aluminum, the desorption enthalpy decreases to 65–70 kJ/mol H₂ (compared to ~ 75 kJ/mol for the pure Mg–H system). This results in a shift of the hydrogen release temperature threshold into the range of 150–280 °C and a noticeable improvement in desorption kinetics [10, 11].

Nevertheless, even the use of such metastabilized alloys does not fully eliminate the fundamental thermodynamic–kinetic dualism and ultimately imposes strict limitations on the choice of metallic materials and the methods by which they can be hydrogenated.

2. Modern approaches to solving the existing problem and substantiating the effectiveness of the hydro-vacuum method

Based on current scientific knowledge and accumulated practical experience, a wide range of methods has been developed to address the aforementioned problem. For example, modern catalytic approaches [12] demonstrate high efficiency in improving hydrogenation kinetics; however, the use of noble metals (Pt, Pd) is limited by their high cost and susceptibility to deactivation due to oxidation and impurity poisoning, which reduces the cyclic durability of such metal-based systems.

Mechanical alloying [13] provides high dispersion (< 50 μm) and stable formation of defect-rich states, but requires prolonged processing and is accompanied by powder contamination from wear products of the milling media.

Plasma- arc synthesis [14], in turn, is applicable only to a narrow class of hydride systems, primarily to compounds such as Alane (AlH₃), and is characterized by extreme thermal gradients that complicate control over the phase composition. Other high- energy hydride- forming techniques, such as electro- pulse treatment of solid- phase suspensions in liquid dielectrics or acoustic- cavitation sonoprocessing [15–17], face challenges associated with low process stability and high energy consumption per unit of product, which significantly limits their productivity and scalability.

The combination of the aforementioned limitations and the need to resolve the existing thermodynamic–kinetic contradiction has led to a clear demand for a new- generation technology capable of integrating the stage of metallic powder structure formation with the operation of its hydrogenation. Such a technology is the innovative hydrovacuum atomization (HVA) process developed by the authors of this study [18–20].

The combined atomization–hydrogenation process implemented in the HVA method is based on a recently identified hydrophysical mechanism of condensational sonomorphosis, which is induced and sustained by the thermobaric shock- wave effects of cavitation fluctuations [21]. This process is accompanied by the in-situ incorporation of hydrogen atoms released from molecules within a supercavitating, cumulatively collapsing, and sonopulsating vapor–water mixture stream, into both the crystalline lattices of the atomized metal and energetically favorable activated

intercrystalline regions containing free electrons, resulting in the formation of interstitial covalent bonds, see Fig. 1.

Against the background of existing technological approaches for producing hydrogenated metallic powders, it can be emphasized that the synergistic combination of atomization and hydrogenation offered by the HVA technology represents a unique engineering solution that integrates the advantages of mechanochemical activation and ultrasonic (sonochemical) treatment while mitigating their key drawbacks. The principal synergistic benefit of this method lies in the coupling of deep powder dispersion with in situ hydrogen saturation within a locally vacuumized and non- stationarily cavitating hydrodynamic medium, wherein the energy released during cavitation bubble collapse and the accompanying cumulative microjets not only contribute to material fragmentation or hydrogen liberation from collapsing cavitation cavities, but also induce a directed sonomorphosis of metastable substructures. These substructures are specifically adapted for prolonged reversible cycling, mutually reversible absorption and release of atomic hydrogen, without significant deterioration of the initial hydrogen accommodation capacity.

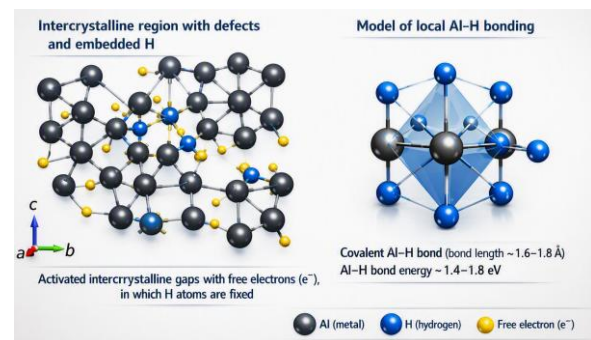


Fig. 1. Physical model of intercrystalline region and local Al–H bonding

The high density of generated crystal structure defects and the continuous exposure of pristine juvenile surfaces free from passivating oxide films ensure unobstructed diffusion and capture of hydrogen deep within the nonequilibrium metallic matrix, which acts as a structure containing numerous hydrogen traps. It should also be noted that an accompanying hydrodynamic Leidenfrost effect [22], arising at the “hot particle–liquid” interface, plays an important role in stabilizing such energetically activated structural states. The quasi- stable vapor envelope temporarily reduces the heat- transfer rate; however, its subsequent explosive collapse generates extreme quenching velocities and continuing intense acoustic- cavitation micro- impacts. These processes inhibit defect relaxation and rigidly fix the metastable, Kirkendall- effect- induced submicron- foamed structure of the powder particles formed during processing (Fig. 2).

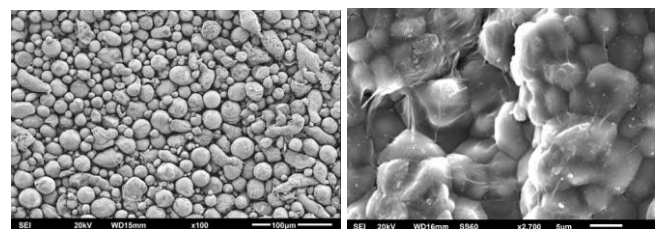


Fig. 2. SEM images of aluminum powder and surface morphology of its particles obtained by hydrovacuum atomization and hydrogenation (Bulk density not exceeding 0.5 g/cm³)

Undoubtedly, a deep understanding of the physical nature of the formation of the described structures and their subsequent controlled engineering, first requires an analysis of the fundamental thermophysical parameters of the hydrovacuum atomization process, with identification of those most critical in terms of their influence on structure formation. This emphasizes the importance

not only of selecting appropriate technological regimes for the target hydrovacuum dispersion process, but also of establishing rational conditions for the preliminary thermophysical preparation prior to feeding the liquid metallic material into the rarefied core of the counter-gravity suction and atomization chamber Fig. 3, position 1.

Consequently, understanding the mutual influence of these factors on the final (operational) characteristics of the powders represents a fundamental basis for improving the conditions of their structural formation and for more effective adaptation to the saturation and solid-state binding of atomic hydrogen.

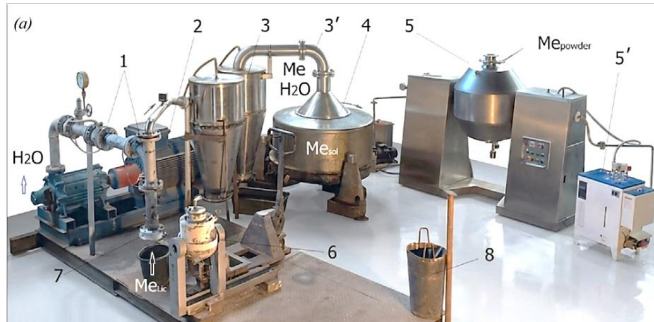


Fig. 3. General view of the hydro-vacuum atomization and hydrogenation installation for metallic melts:

- 1 — Hydro-vacuum suction, dispersion, and melt-cooling chamber (Me_{lic})
- 2 — High-pressure pump for high-velocity water injection into the chamber (water delivery pressure 12–20 bar, flow rate 20–22 L/s)
- 3 — Pair of scrubbers for primary gravitational separation of coarse powder fractions (1–2.5 mm) from the forming slurry (Me)
- 3' — Pipeline for slurry transfer from the scrubbers (3) to the dewatering centrifuge (4)
- 5 — Vacuum drying chamber for dehydrated powder
- 5' — Thermovacuum station of the drying chamber (5)
- 6 — Unit for metered feeding of the melt to the disperser-cooler (1)
- 7 — Vertical melt-suction head
- 8 — Ladle supplying the melt to the feeding unit (6)

Addressing the stated problem requires a predictive assessment of the thermophysical regimes governing the structure formation of the atomized liquid metallic material. Achieving such predictive accuracy becomes feasible through computer simulation of the cooling and quenching processes experienced by the fragmented particles formed during hydro-vacuum atomization.

The application of numerical computer modeling makes it possible, in an inverse formulation, to determine the ranges of initial (input) parameters for melt preparation that are most closely aligned with the optimal conditions required for the target hydro-vacuum treatment. In this way, modeling functions as a tool that provides a closed optimization loop: from forecasting the structural states of the atomized particles to adjusting the initial technological conditions that ensure the desired properties of the final material.

3. Factors and assumptions taken into account in the simulation

1) The interaction of a high-energy water jet (velocity 200 m/s) and the melt occurs by dispersing the latter into spherical droplets with a diameter of 10–1000 μm , the cooling of which is carried out in the presence of a gasified film formed as a result of the Leidenfrost effect; 2) Under the influence of the impact force developed during the spraying of water in the disperser, the vapor film is expected to degrade and be completely removed from the surface; 3) The temperature field in the atomized metal is distributed homogeneously $T_{liq}(t)=\text{const.}$; 4) The heat exchange process between the metal and water includes both convective and radiative (thermal radiation) components; 5) The melt solidifies at a fixed temperature $T_m=T_{lic}=T_{sol}$.

Modeling and its results: For a spherical particle with an average radius r , the volume (V), mass (m) and surface area (A) are defined as follows:

$$V = (4/3) \pi r^3, m = \rho V, A = 4\pi r^2.$$

However, considering that the metal melt is dispersed into N equal number of droplet fragments so that the total mass remains unchanged, the effective surface area increases approximately as follows: $A_{eff} = AN^{1/3}$. Then, the heat radiation intensity (q_{dot} , W) from the metal fragments with a temperature T_{liq} and a velocity v will be:

$$q_{dot} = A_{eff} [h(T_{f,v})(T_{liq} - T_w) + \varepsilon \sigma ((T_{liq} + 273.15)^4 - (T_w + 273.15)^4)] \quad (1)$$

Where, $h(T_{f,v})$ -convective heat transfer coefficient of the boundary layer, depending on the effective temperature of the cooling medium (water/steam) ($T_{film}=0.5(T_{liq}+T_w)$, $W/(m^2 \cdot K)$; T_{liq} - temperature of the metal entering the dispersion chamber, $^{\circ}\text{C}$; T_w - temperature of the dispersing and cooling water, $^{\circ}\text{C}$; ε - blackness of the metal surface (emissivity); σ - Stefan-Boltzmann constant, $5.670374419 \times 10^{-8} W/(m^2 \cdot K^4)$; v - water jet velocity, m/s.

To account for the specific heat capacity (c_v , J/kg·K) of the boundary layer, the computer script uses the following simple approximation:

$$h(T_{f,v}) = \min(h_{base} + c_v \sqrt{v}, h_{max}) \quad (2)$$

Where, h_{base} is the base heat transfer coefficient under stationary conditions, and h_{max} is the maximum allowable limit (upper limit) of the coefficient. For the case of Leidenfrost stability, we obtain the following:

$$h(T_{f,v}) = 8000 + 600 \sqrt{200} \approx 16485 \quad (3)$$

When the vapor barrier degrades (breaks), the coefficient takes on the following value:

$$h(T_{f,v}) = 25000 + 600 \sqrt{200} \approx 33485 \quad (4)$$

In addition, the calculation model for the cooling rate of the metal is based on enthalpy balance and, taking into account its specific heat capacity $c_{p,liq}$ and the condition $T_{liq} > T_{sol}$, takes the following form:

$$m c_{p,liq} dT/dt = -q_{dot} \Rightarrow dT/dt = -q_{dot} / (m c_{p,liq}) \quad (5)$$

In expanded form, the heat transfer equation is formed as the following mathematical representation:

$$dT/dt = -A_{eff} [h(T_{f,v})(T_{liq} - T_w) + \varepsilon \sigma ((T_{liq} + 273.15)^4 - (T_w + 273.15)^4)] / (m c_{p,liq}) \quad (6)$$

We assume that the actual temperature of the liquid metal T_{liq} remains unchanged until the latent heat of crystallization L_f (J/kg) is completely released from the cooling fragments. That is, the metal completely solidifies when the current latent heat supply becomes zero ($L_{(t)} = 0$):

$$T = T_{sol}, T_{liq, Initial} = m L_f, dL/dt = -q_{dot}, dT/dt = 0 \quad (7)$$

After the latent heat of crystallization is completely released $L(t) = 0$ and the prerequisite for further cooling of the solid phase $T < T_{sol}$ is created, the subsequent rate of temperature change is described by the following differential equation:

$$m c_{p,sol} dT/dt = -q_{dot}, \text{ from where: } dT/dt = -q_{dot} / (m c_{p,sol}) \quad (8)$$

Where, $c_{p,sol}$ is the specific heat capacity of the metal in the solid state. And dT/dt is calculated in a similar way to the expanded heat transfer equation (6), by replacing the heat capacity of the liquid phase in it with the solid.

4. Results and discussion

The obtained modeling results, specifically t_{sol} (cooling time to the solidus temperature T_{sol}) and $t_{100^\circ C}$ (cooling time to the boiling point of water, $100^\circ C$) — were obtained for atomized particles of lead, aluminum, magnesium, Armco- iron, and Al–Mg alloys. The simulations were performed using the initial thermophysical and thermodynamic parameters (ρ , T_{lic} , $C_{p,liq}$, $C_{p,sol}$, L_f , and T_{liq}) listed in Table 1.

The resulting cooling- kinetics contour maps, shown in Figure 4, illustrate the temperature evolution of particles of different diameters during hydrovacuum atomization. These cooling kinetics maps (isotherms) reveal distinct cooling behaviors governed by the intrinsic thermal properties of each material. In particular:

Lead (Pb) demonstrates the slowest cooling due to its high density and relatively low thermal diffusivity.

Aluminum (Al) cools significantly faster, with steep temperature gradients even for larger particle diameters.

Magnesium (Mg) exhibits the most rapid cooling, driven by its low density and high thermal conductivity.

Armco- iron (Fe) shows prolonged high- temperature retention, reflecting its high melting point and substantial latent heat of fusion.

Al–Mg alloys display intermediate cooling kinetics, with the 30%Al – 70%Mg and 70%Al – 30%Mg compositions forming distinct thermal- response profiles.

Table 1

Met.	ρ , kg/m ³	T_{lic} , °C	$C_{p,liq}$, J/kg·K	$C_{p,sol}$, J/kg·K	L_f , x10 ³ , J/kg	T_{liq} , °C
Pb	10660	327.5	160	128	23	370
Al	2375	660.3	1170	900	397	700
Mg	1570	650.0	1370	1020	370	700
Fe	6980	1538	820	450	247	1600

These cooling- time fields directly determine the structurogenesis pathways of atomized droplets. The combination of particle diameter and cooling rate defines whether the droplets undergo: full crystallization, partial solidification with retained liquid cores, formation of fine- grained or amorphous structures, development of dendritic, cellular, or equiaxed morphologies, suppression or activation of diffusion- controlled transformations, hydrogen uptake and defect stabilization during hydrovacuum processing.

Comparative analysis of the modeled thermal processes shows that, following the primary influence of particle dispersity (their diameter, d) on the cooling rate, the next most significant factors are the thermophysical properties of the material and the initial temperature of the melt supplied to the hydrovacuum disperser.

Analysis of the time required for the particles to reach the boiling point of water ($100^\circ C$) reveals several key regularities:

- Effect of particle size:

For all investigated materials, the fine fraction with a diameter of $d = 0.1$ mm ($100\ \mu m$) cools to $100^\circ C$ approximately ten times faster than larger particles with $d = 1.0$ mm ($1000\ \mu m$). This behavior is governed by the increase in specific surface area as particle size decreases.

- Armco- Fe exhibits the highest cooling rate among all materials considered. Due to its high initial temperature ($1530^\circ C$) and the intense radiative-convective heat transfer, a particle with a diameter of 0.1 mm cools to $100^\circ C$ in only 0.006 s (average cooling rate $2.38 \times 10^5\ ^\circ C/s$), whereas a particle of 1.0 mm reaches $100^\circ C$ in 0.062 s (average cooling rate $2.3 \times 10^4\ ^\circ C/s$).

- Magnesium (Mg) and aluminum (Al), despite having nearly identical initial temperatures ($720^\circ C$), exhibit different cooling characteristics. Specifically, magnesium particles cool faster (for $d = 0.1$ mm: $t \approx 0.007$ s, $v \approx 8.86 \times 10^4\ ^\circ C/s$) than aluminum particles ($t \approx 0.009$ s, $v \approx 6.89 \times 10^4\ ^\circ C/s$). This difference is attributed to the lower density and caloric properties of magnesium.

- In Al–Mg alloy systems, an increase in the magnesium content leads to an intensification of the cooling process.

- For the alloy containing 30% Al – 70% Mg, the cooling time of a 0.1 mm particle down to $100^\circ C$ is $t \approx 0.008$ s ($v \approx 7.75 \times 10^4\ ^\circ C/s$), whereas for the alloy with 30% Mg – 70% Al this time increases to $t \approx 0.010$ s ($v \approx 6.20 \times 10^4\ ^\circ C/s$).

- For lead (Pb), due to its comparatively low initial temperature ($370^\circ C$), the average cooling rate varies from $5.4 \times 10^4\ ^\circ C/s$ (for $d = 0.1$ mm, $t \approx 0.005$ s) to $5.19 \times 10^3\ ^\circ C/s$ (for $d = 1.0$ mm, $t \approx 0.052$ s).

Analysis of the obtained results shows that the solidification rate (the $Me_{liq} \rightarrow Me_{sol}$ phase transition) is primarily governed by the latent heat of crystallization, the specific heat capacity, and the initial thermal interval — i.e., the degree of superheating ($T_{liq} - T_{lic}$). For example, although the crystallization temperature of Armco iron is nearly twice that of aluminum, and although aluminum is less superheated during dispersion compared to Armco iron, its solidification duration is nevertheless longer (0.03312 s vs. 0.02736 s). This behavior is explained by the fact that the latent heat of aluminum ($397\ 000$ J/kg) is significantly higher than that of iron ($247\ 000$ J/kg), and its specific heat capacity in the solid state ($C_{p,sol}$) is approximately twice that of iron.

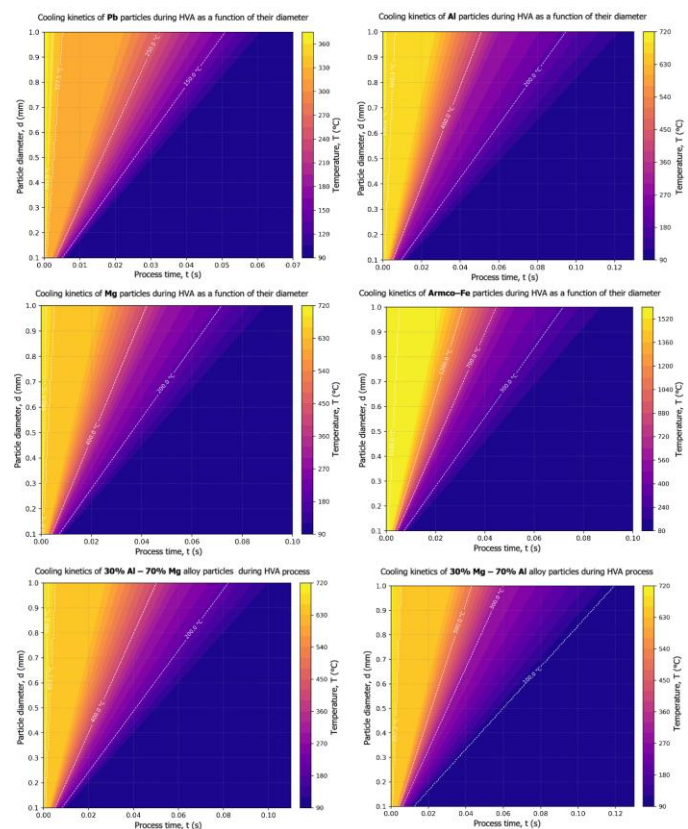


Fig. 4. Modeling results: Dependence of the cooling rate of hydrovacuum- atomized particles of lead, aluminum, magnesium, Armco-Fe, and Al–Mg alloys on dispersion duration and particle size - fraction 100–1000 μm .

In turn, analysis of the diagrams shown in Fig. 5 indicates that, due to the reduction of powder particle size to 0.01 mm ($10\ \mu m$) — and, consequently, the increase in the surface- area- to- mass ratio (A/m) — the cooling rates in this range rise sharply and reach values on the order of $10^5\ ^\circ C/s$.

In particular, analysis of the time required for particles to reach $100^\circ C$ for the extreme boundaries of the fraction ($d = 0.01$ mm ($10\ \mu m$) and $d = 0.10$ mm ($100\ \mu m$)) leads to the following findings:

For aluminum particles with a size of 0.01 mm ($10\ \mu m$), the cooling time from the initial melt temperature ($\approx 720^\circ C$) down to $100^\circ C$ is less than 0.002 s (approximately 1.8 – 2.0 ms). The

corresponding average cooling rate exceeds $6.5 \times 10^5 - 7.7 \times 10^5$ °C/s, which is characteristic of ultra- rapid solidification conditions.

For aluminum particles with a size of 0.10 mm (100 μ m), the time required to reach 100 °C is approximately 0.0125 s, and the average cooling rate is about 5.3×10^4 °C/s.

The 30% Mg – 70% Al alloy exhibits cooling dynamics similar to those of pure aluminum: a 0.10 mm particle cools to 100 °C in 0.012 s, with an average cooling rate of $\approx 5.5 \times 10^4$ °C/s.

In the 50% Al – 50% Mg alloy, due to reduced density and modified heat capacity, the cooling time of a 0.10 mm particle decreases to 0.0105 s (cooling rate $\approx 5.9 \times 10^4$ °C/s).

In turn, the 70% Mg – 30% Al alloy demonstrates the highest cooling rate within the investigated particle- size fraction: a 0.10 mm particle reaches 100 °C in 0.0115 s, with an average cooling rate of $\approx 6.4 \times 10^4$ °C/s, which is attributed to the predominance of the magnesium matrix with lower specific density.

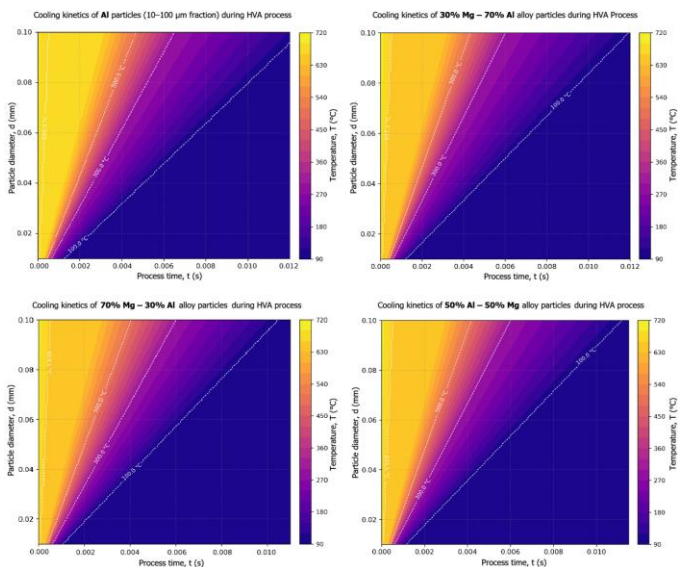


Fig. 5. Modeling results: Dependence of the cooling rate of hydrovacuum-atomized particles of aluminum and Al-Mg alloys on dispersion duration and particle size - fraction 10–100 μ m.

In all cases, a reduction in particle diameter within the investigated range from 100 μ m to 10 μ m leads to an increase in cooling rate by approximately a factor of 10–12.

This allows us to conclude that using the 10–100 μ m fraction during hydrovacuum atomization of Al-Mg alloys enables the stable attainment of ultra- high cooling rates on the order of $10^4 - 10^5$ °C/s, thereby creating favorable conditions for the formation of fine- crystalline, highly heterogeneous, metastabilized substructures in the resulting powders.

This trend is confirmed by the experimental data presented in Fig. 6.

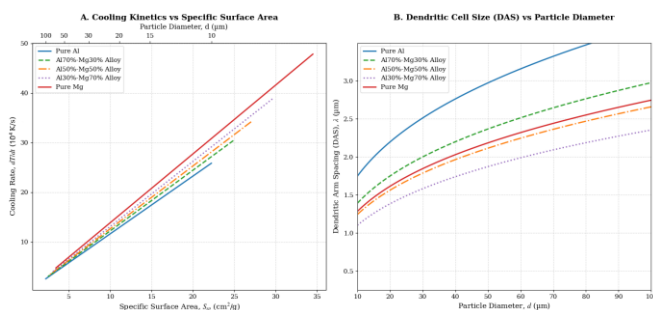


Fig. 6. Influence of the specific surface area of powder particles and their diameter on the cooling kinetics and the size of the resulting dendritic cells.

As seen in Fig. 6, a reduction in particle size from 100 μ m to 10 μ m and an increase in specific surface area from 2–3 to 25–35 cm²/g lead to a pronounced rise in cooling rate (diagram A), which in turn results in a refinement of the dendritic cell size from 2.5–3.0 μ m down to 1.2–1.6 μ m (diagram B).

In binary Al–Mg alloys (particularly in compositions Al 50% – Mg 50% and Al 30% – Mg 70%), the combined effects of constitutional supercooling and segregation processes produce a more dispersed dendritic structure compared to the pure components.

For fine powder fractions ($d < 50$ μ m), the formation of an ultra- fine microstructure (< 2.5 μ m) is reliably ensured, which predetermines a high degree of thermodynamic nonequilibrium and metastable structural state in the resulting powders.

Quenching from the liquid state stabilizes metastable phases and supersaturated solid solutions characterized by a high level of excess internal energy (Gibbs energy).

This substantially reduces both the thermodynamic and kinetic barriers for hydrogen–material interaction, lowering the activation energy of chemisorption.

The extreme cooling kinetics generate a high- density defect structure (high concentrations of vacancies, dislocations, and microstrains).

Local lattice distortions facilitate the incorporation of hydrogen atoms into tetrahedral and octahedral interstitial sites, while microscale segregation and phase heterogeneity create localized regions with increased chemical affinity toward hydrogen.

As a result, hydrogen absorption proceeds with high kinetics at reduced temperatures and pressures. Moreover, in the case of repeated hydrogenation of powders previously dehydrogenated by conventional methods, no prolonged high- temperature, mechanical, or catalytic activation is required, since the metastable structural state remains preserved.

A comprehensive, integrated analysis of the obtained data shows that the key factor governing the kinetics of the phase transition and the resulting metastabilized morphogenesis of powder particles is the coupled influence of the latent heat of crystallization (L_f), which depends on density (ρ), and the melt superheating ($\Delta T = T_{liq} - T_{lic/sol}$), which in turn is determined by the actual melt preparation temperature (T_{liq}) prior to the onset of HVA.

For example, according to numerical thermodynamic modeling, the anomalously high latent heat of crystallization of aluminum ($L_f = 397\,000$ J/kg), combined with the elevated specific heat capacity of its solid phase ($c_{p,sol} = 900$ J/(kg·K)), substantially prolongs the overall solidification duration of a droplet compared with other metallic systems.

In particular, when compared with a magnesium matrix, which possesses a similar latent heat ($L_f = 370\,000$ J/kg) and even higher specific heat capacity ($c_{p,sol} = 1020$ J/(kg·K)), the calculated time required for complete cooling of aluminum particles down to 100 °C is significantly longer ($t_{100^\circ C} = 0.12345$ s vs. 0.08182 s for magnesium, Fig. 4).

This behavior is explained by the substantial difference in density between the compared molten metals (2375 kg/m³ vs. 1570 kg/m³). The higher density of aluminum results in a proportionally greater droplet mass at an equal diameter d (mm), which leads to a significantly larger volumetric heat- storage capacity ($\rho \cdot L_f$ and $\rho \cdot c_p$).

As a consequence, the specific surface area available for heat removal per unit mass ($A/m = 4\pi r^2 / \rho V$) is lower for aluminum, which ultimately prolongs both the phase- transition processes and subsequent cooling. This trend is reflected in Fig. 6A (blue curve).

Additionally, the melt- superheating factor (ΔT) makes a substantial contribution.

A high melt- preparation temperature T_{liq} generates a significant excess of thermal energy in the liquid phase ($Q_{overheat} = m \cdot c_{p,liq} \cdot \Delta T$), which must be dissipated before the onset of solidification.

In combination with the high volumetric heat capacity of aluminum ($\rho \cdot c_p$) and its latent heat (L_f), this leads to an overall

increase in the thermal inertia of the droplet and explains the pronounced prolongation of the time required for complete cooling ($t_{100^\circ\text{C}}$). Consequently, aluminum exhibits a comparatively coarse dendritic structure (cell sizes $> 3 \mu\text{m}$, Fig. 6B, blue curve).

On the other hand, the large magnitude of the phase enthalpy reflected in the latent heat of crystallization (L_f), when combined with extreme convective–radiative heat extraction, creates unique conditions for the quasi- instantaneous fixation of strongly nonequilibrium substructures.

The latent heat released during aluminum crystallization briefly maintains high matrix plasticity within the temperature interval of the phase transition. Against the background of continuous ultrasonic shock- wave loading from the HVA medium, this promotes the intensive development of shear deformations, the generation of dislocation ensembles, and the stabilization of a pronounced “spongy” porosity throughout the volume of the forming powder particle (Fig. 7).

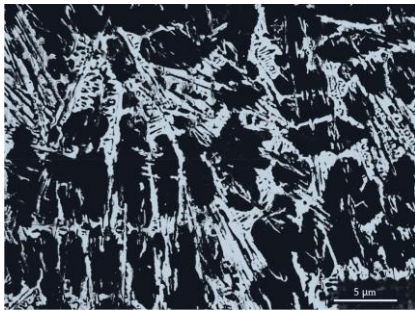


Fig. 7. Microsection of a particle produced by hydrovacuum atomization from the Al–Mg alloy (Al 70% – Mg 30%), particle from the 100 μm fraction.

Since, in the HVA process, the primary adjustable parameter governing the cooling (and, accordingly, supercooling) regime is the degree of initial superheating of the melt supplied for atomization, it can be stated that targeted control of this parameter enables regulation of the actual residence time of the dispersed fragment in both the liquid and the transitional liquid–crystalline states. This residence time is critically important for structural formation, activation, and the degree of hydrogenation.

It is noteworthy that, in the case of Al–Mg and other binary alloys possessing a defined solidification interval, an equally important control criterion becomes the establishment of an optimal balance between the melt- superheating level and the temperature range (interval) of crystallization (as determined from phase diagrams).

As demonstrated by the experimental results, this factor acts as the key technological lever in the combined processes of structure formation and hydrogenation (Figs. 8 and 9).

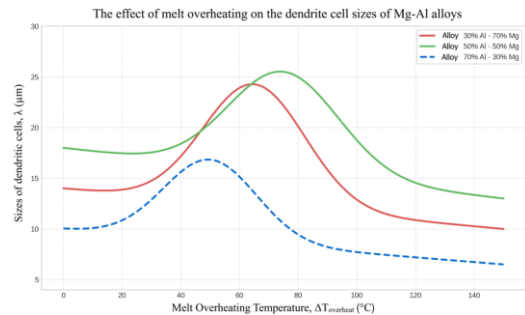
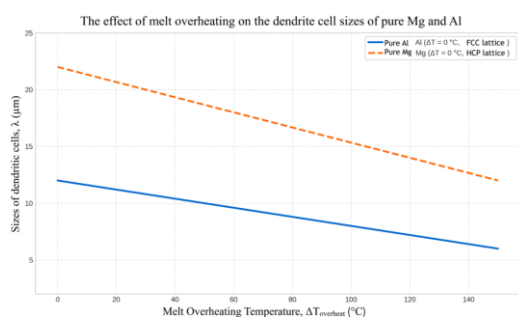


Fig. 8. Influence of melt superheating on the size of dendritic cells.

High melt superheating reduces melt viscosity and surface tension, which, under hydrovacuum atomization, ensures finer droplet fragmentation and an increased cooling rate.

Since pure metals do not exhibit complex eutectic transformations or a dendrite- forming interval ($T_{lic} \approx T_{sol}$), a simple direct inverse relationship is observed: - the higher the superheating, the finer the resulting structure.

Pure magnesium, due to the characteristics of its hexagonal close- packed lattice and its stronger tendency for directional crystal growth, initially exhibits a coarser grain size than pure aluminum with its face- centered cubic lattice; however, the overall trend of the curves for both metals remains descending.

In contrast to pure metals, binary Al–Mg alloys possess a wide solidification interval ($\Delta T > 0$), which is determined from the phase diagram.

Within the range of moderate superheating (typically around 50–80 $^\circ\text{C}$), the curve shows a pronounced rise, - an increase in the average grain size (the peak region).

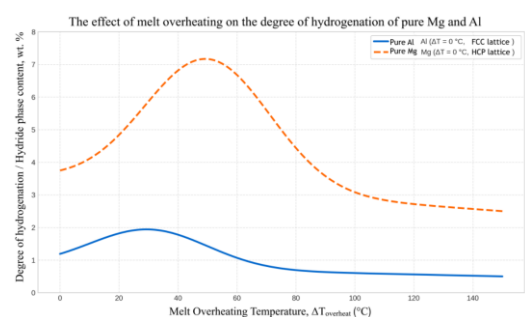
This behavior is associated with the persistence of nucleating microclusters in the melt and enhanced liquation (component segregation), both of which promote the coarsening of dendritic structures during solidification.

Compositions enriched in magnesium or located near the eutectic exhibit a wider solidification interval, which makes the grain- coarsening peak more pronounced.

The aluminum- rich alloy (70% Al, 30% Mg — blue dashed line) has a narrower solidification interval and correspondingly smaller grain- size values.

Notably, when the melt- superheating temperature exceeds a critical threshold (100–120 $^\circ\text{C}$), all inherited micro- inhomogeneities and clusters are completely destroyed.

The melt becomes fully homogeneous, which under subsequent rapid cooling allows the solidification interval to be traversed with suppressed grain growth, ensuring the formation of minimal grain sizes in the final powder.



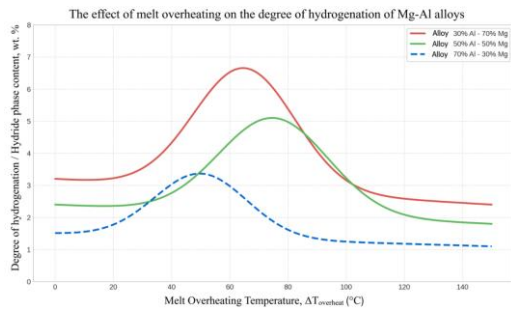


Fig. 9. Influence of melt superheating on the degree of hydrogenation.

Alloys with high magnesium content (70%) exhibit the strongest tendency to interact with hydrogen and to form metastable MgH_2 phases, showing the highest hydrogenation degrees. Alloys with 50% Mg contain a well- developed eutectic component and a highly defective microstructure at moderate superheating, forming a pronounced peak. Aluminum- rich alloys (30% Mg), due to the partial protective influence of finely dispersed nanoporous aluminum- oxide films, demonstrate the lowest hydrogenation values.

However, aluminum is not a passive component; on the contrary, it actively participates in hydrogen chemisorption, forming surface Al-H complexes and promoting hydrogen redistribution within the solid phase.

The thermodynamic stability of aluminum and its higher Al-H bond energy partially "counterbalance" the excessive reactivity of magnesium, weakening Mg-H interactions and facilitating reversible low- temperature dehydrogenation, - a particularly important factor for cyclic processes.

This behavior is confirmed by the X- ray diffraction data illustrated in Fig. 10.

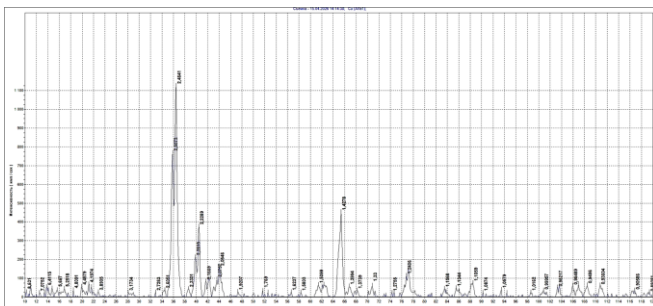


Fig. 10. X- ray diffraction pattern of the atomized Al-Mg alloy powder (70% Mg – 30% Al).

The presented X- ray diffraction pattern shows that the investigated sample exhibits well- defined, intense diffraction peaks corresponding to the coexistence of crystallographic phases of the initial metallic components (magnesium and aluminum), as well as the products of their interaction with hydrogen.

The presence of distinct reflections confirms the formation of the magnesium hydride phase MgH_2 , along with stable intermediate structures and surface Al-H complexes, which directly correlate with hydrogen redistribution processes within the solid phase.

The presence of characteristic peaks with well- defined interplanar spacings reflects the high degree of dispersity and defectiveness of the crystalline structure, which is governed by the developed eutectic component and the influence of protective nanoscale oxide films.

These structural features subsequently reduce the overall energy required for reversible activation and enable low- temperature reversible dehydrogenation of the alloy.

In particular, magnesium constitutes the primary component of the matrix structure. The most intense analytical peak is recorded at $2\theta = 36.43^\circ$ ($d = 2.4641 \text{ \AA}$, relative intensity 100%, contributing ~21.78% to the volumetric phase content). High- angle magnesium reflections are observed at $2\theta = 106.95^\circ$ ($d = 0.9585 \text{ \AA}$), 108.41° ($d = 0.9496 \text{ \AA}$), and 110.75° ($d = 0.9360 \text{ \AA}$).

Aluminum, in turn, participates in forming the structural framework of the alloy. A characteristic aluminum peak is detected at $2\theta = 65.29^\circ$ ($d = 1.4278 \text{ \AA}$, intensity 288 imp/s, mass fraction ~12.85%), as well as at $2\theta = 38.61^\circ$ ($d = 2.3301 \text{ \AA}$).

The intermetallic compounds $\text{Al}_{12}\text{Mg}_{17}$ and AlMg_3 represent products of alloying and eutectic solidification. The $\text{Al}_{12}\text{Mg}_{17}$ phase is identified at $2\theta = 65.29^\circ / 88.29^\circ$ ($d = 1.1059 \text{ \AA}$), while AlMg_3 appears at $2\theta = 21.15^\circ$ ($d = 4.1974 \text{ \AA}$).

The presence of these phases confirms the well- developed eutectic structure of the alloy.

Diffraction peaks corresponding to AlH_3 are registered in the medium- and high- angle regions at $2\theta = 41.64^\circ$ ($d = 2.1669 \text{ \AA}$), 52.55° ($d = 1.7400 \text{ \AA}$), 61.35° ($d = 1.5099 \text{ \AA}$), and within the $83\text{--}106^\circ$ range ($2\theta = 83.49^\circ, 85.54^\circ, 93.45^\circ, 105.93^\circ$).

This confirms that aluminum is not inert but actively participates in hydrogen chemisorption, forming surface Al-H complexes.

Characteristic reflections of orthorhombic MgH_2 (typically strong peaks at $2\theta \approx 27.9^\circ, 35.7^\circ, 38.2^\circ$, and 39.9°) appear near the magnesium peaks; however, in the experimental diffractogram they merge into broadened composite peaks due to the nanoscale grain state, — high defect density and small crystallite thickness ($17\text{--}19 \text{ nm}$).

The recorded significant peak half- width ($0.4639^\circ \approx 0.0081 \text{ rad}$) combined with high intensity ($I = 734 \text{ imp/s}$) indicates not amorphization but pronounced subgrain fragmentation and a high dislocation density, both induced by the extreme thermal gradients inherent to hydro-vacuum atomization.

Hydroxide-hydride complexes ($\text{AlH}_3 \cdot \text{O}_3$) are also present in the diffractogram, appearing in the low- angle region at $2\theta = 13.80^\circ$ and 14.73° , reflecting the specific surface interactions of moisture/oxygen with the forming hydride structures.

Notably, the intermetallic $\text{Al}_{12}\text{Mg}_{17}$ reflection at $2\theta = 65.29^\circ$ ($d = 1.4278 \text{ \AA}$) exhibits a peak half- width of 0.6971° , corresponding to an ultrafine crystallite size of approximately $14\text{--}15 \text{ nm}$.

For the oxide phase periclase (MgO), the half- width at $2\theta = 35.78^\circ$ is 0.4407° , while for Al_2O_3 at $2\theta = 39.83^\circ$ it is 0.4272° .

These small coherent- scattering domain sizes confirm the formation of protective nanoporous oxide frameworks.

The maximum peak broadening is observed for reflections at $2\theta = 16.87^\circ$ (0.8080° for $\text{Al}_{12}\text{Mg}_{17}$), $2\theta = 62.35^\circ$ (0.9210° for Mg), and $2\theta = 77.07^\circ$ (0.7190°).

Such pronounced broadening at low and medium intensities indicates a high level of type- II microstrains within the crystal lattice, caused by local lattice distortions due to incorporated hydrogen atoms and vacancies.

Thus, the dependencies of λ and the hydrogenation degree on $\Delta T_{\text{overheat}}$, presented in Figs. 8 and 9, consistently demonstrate that the thermodynamically governed evolution of the dendritic- structure scale is the key factor determining the hydrogen- saturation capacity and functional properties of powders produced by hydrovacuum atomization.

5. Generalized semi- empirical model for the formation of crystalline- structure parameters.

Based on the theoretical and experimental data obtained in this study, a semi- empirical generalized expression was developed to mathematically describe the combined influence of melt- superheating temperature and the solidification interval on the resulting structural scale (dendritic- cell or grain size, λ).

The model incorporates three principal factors: 1) Baseline structural refinement driven by increasing superheating ($\Delta T_{\text{overheat}}$); 2) Structural coarsening in the moderate- superheating region, associated with the persistence of micro- inhomogeneities and the

width of the solidification interval ($\Delta T_{\text{interval}}$); 3) An individual component- affinity coefficient, reflecting the tendency of alloy constituents to form clusters or protective films, - in this case, governed by the Al/Mg ratio.

$$\lambda = \lambda_0 \cdot \exp(-\Delta T_{\text{overheat}}/k_1) + A_1 \cdot (\Delta T_{\text{interval}}/T_{\text{ref}}) \cdot \exp(-(\Delta T_{\text{overheat}} - T_{\text{crit}})^2 / 2 \sigma^2) + C \quad (9)$$

Where: λ - resulting dendritic- cell or grain size (μm); λ_0 - initial structural scale of the base pure metal; $\Delta T_{\text{overheat}}$ - melt- superheating temperature above the liquidus ($^{\circ}\text{C}$); $\Delta T_{\text{interval}}$ - alloy solidification interval ($T_{\text{liquidus}} - T_{\text{solidus}}$), defining the width of the phase- transition zone ($^{\circ}\text{C}$); k_1 - thermal- damping coefficient (responsible for monotonic cell narrowing under deep superheating); A_1 - coefficient describing the influence of liquation and inherited clusters (directly proportional to the fraction of components with a wide solidification interval, e.g., 30% Al–70% Mg or 50% Al–50% Mg compositions); T_{crit} - critical moderate- superheating temperature (typically 50–80 $^{\circ}\text{C}$) at which the structural peak (melt heterogeneity) is most pronounced; T_{ref} - reference normalization temperature (100 $^{\circ}\text{C}$); σ - width of the melt- instability peak; C - technological limit of structural refinement under extremely high cooling rates during hydrovacuum dispersion.

The proposed generalized relationship makes it possible to quantitatively describe how the thermodynamic properties of metallic materials influence structure formation during hydrovacuum atomization.

Specifically: – the first exponential term reflects the dominance of thermodynamic factors; as the melt- superheating temperature increases ($\Delta T_{\text{overheat}} \gg k_1$), its viscosity and surface tension decrease exponentially, leading to a monotonic reduction in the dendritic- cell scale (λ).

– the second term, a Gaussian bell- shaped function, describes the “anomalous” structural peak that arises within the moderate- superheating range (T_{crit}), where liquation- induced heterogeneities and cluster groupings persist.

The amplitude and width of this peak are governed by the solidification interval ($\Delta T_{\text{interval}}$): it vanishes for pure metals ($\Delta T_{\text{interval}} \rightarrow 0$), while in Al–Mg alloys it produces a pronounced maximum.

Equation (9), together with the experimental trends shown in Figs. 8 and 9, demonstrates a direct correlation between the dendritic- structure scale λ and the hydrogen- saturation capacity of the particles. For pure metals (Al, Mg), the monotonic decrease in λ with increasing $\Delta T_{\text{overheat}}$ (Fig. 8) leads to a higher fraction of defective boundaries and active surface sites, which manifests as an increased degree of hydrogenation and a higher content of hydride phases (Fig. 9). This effect is especially pronounced for magnesium and Mg- rich systems.

In Al–Mg alloys, the nonlinear, hump- shaped dependence $\lambda(\Delta T_{\text{overheat}})$, governed by the Gaussian term in Equation (9), leads to the fact that within the intermediate- superheating region ($T_{\text{crit}} \approx 50\text{--}80\text{ }^{\circ}\text{C}$), dendritic- cell coarsening is accompanied by a reduction in the effective area of active boundaries and a decrease in the hydride- phase fraction.

Upon transitioning to deep superheating ($>120\text{ }^{\circ}\text{C}$) followed by rapid cooling, λ decreases again, and the structure becomes fine- dispersed and defect- enriched.

This promotes enhanced hydrogen diffusion and uptake, resulting in higher hydrogenation degrees, - particularly in Mg- rich compositions.

According to the derived equation, the resulting dendritic- cell or grain size, being itself dependent on the solidification duration of the particles, directly influences the depth and character of hydrogen uptake. Consequently, the longer a particle remains within the high- temperature transitional solidification interval, the more time is available for sonovolumetric action and condensational sonomorphosis, both of which arise under hydrovacuum

atomization conditions. These effects enhance hydrogen diffusion and capture, stabilize defect- rich states, and generate activated structures capable of sustaining multiple hydrogenation–dehydrogenation cycles.

Taken together, these findings lead to the key conclusion that the thermodynamics of phase transformations, alloy composition, and solidification kinetics jointly determine the efficiency of structure formation and its targeted adaptation to subsequent hydrogenation–dehydrogenation cycles.

This establishes a fundamental basis for developing methods of purposeful design of highly stable functional powders for hydrogen- energy applications.

6. Conclusion

The analysis of the thermodynamic properties of metallic materials, combined with thermal- regime modeling of particle cooling during hydrovacuum atomization, demonstrates that differences in heat capacity, thermal conductivity, and latent heat of fusion generate unique temperature–time cooling trajectories for the produced particles, thereby defining their structural state.

The modeling results, together with data from parallel experimental investigations, establish a closed optimization loop for the technological process: thermodynamic properties $\rightarrow$ thermal history of particles $\rightarrow$ structure formation $\rightarrow$ hydride formation $\rightarrow$ adjustment of melt parameters $\rightarrow$ increased efficiency of atomization and hydrogenation.

This approach enables predictive control over the structural evolution and reactive capability of atomized metallic systems, providing a scientifically grounded platform for designing highly efficient regimes of hydrovacuum processing and hydrogenation.

The constructed thermal maps (isotherms) of particle cooling demonstrate that the parameters t_{sol} and $t_{100^{\circ}\text{C}}$ serve as reliable indicators of solidification and cooling kinetics and can be used to predict the critical intervals of dendritic, highly activated, and therefore hydrogen- intensive structure formation.

It was found that rapidly cooling systems (Mg, Al–Mg alloys) tend to form fine- dispersed, highly defective, and metastable structures, whereas materials with high thermal inertia (Pb, Armco- Fe) retain coarse structural elements and prolonged high- temperature states. This explains their lack of competitiveness compared to the lightweight metals investigated in this study.

It is shown that the reactive (chemisorptive) hydrogen- uptake capability is determined not only by composition but also by the combined thermodynamic properties of the alloy components.

Mg- rich systems (50–70% Mg) indeed exhibit a strong tendency toward hydride formation (MgH_2). However, aluminum, when present alongside magnesium, does not act as a passivating component; rather, it actively participates in hydrogen chemisorption, forming surface Al–H complexes and facilitating hydrogen redistribution within the solid phase.

The thermodynamic stability of aluminum and its higher Al–H bond energy partially offset the excessive reactivity of magnesium, attenuating Mg–H interactions and enabling reversible low- temperature dehydrogenation, a feature of particular importance for cyclic hydrogenation–dehydrogenation processes.

Two scientifically substantiated pathways for the purposeful production of HVA- powders for different applied tasks have been identified.

For obtaining dense, homogeneous particles of globular morphology, eutectic and near- eutectic compositions are optimal, as they ensure the condition $\Delta T_{\text{interval}} \rightarrow 0$, maximize the solidification rate, and minimize the free energy of the system.

For generating metastable, activated structures adapted to repeated hydrogenation–dehydrogenation cycles, alloys with a wide solidification interval ($\Delta T_{\text{interval}} \neq 0$) are preferable, provided that the melt- superheating level $\Delta T_{\text{overheat}}$ is strictly controlled and satisfies the condition $\Delta T_{\text{overheat}} \geq \Delta T_{\text{interval}}$ during the preparation stage prior to hydrovacuum atomization.

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