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STUDY OF THE STRUCTURAL-PHASE STATE OF THE MECHANICALLY ACTIVATED SYSTEM Pd-Ti-Mg

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This work examines the structural–phase state and microstructural organization of mechanically activated ternary Pd-Ti-Mg powders as candidate materials for solid-state hydrogen storage. Elemental powders were processed by high-energy planetary milling (Pulverisette 7, 500 rpm; BPR=10:1) in Ar. X-ray diffraction (Cu Kα) reveals fcc-Pd (Fm-3m, a=3.887 Å), α-Ti (hcp, $P6_3/mmc$, a=2.950 Å, c=4.681 Å), a metastable fcc-Ti fraction (Fm-3m, a=4.060 Å), and minor hcp-Mg (P63/mmc, a=3.209 Å, c=5.211 Å); no distinct intermetallic peaks are resolved at the bulk level. Laser diffraction shows a multimodal size distribution with D10≈5 μm, D50≈20 μm, D90≈50 μm (span≈2.25), a fine sub-5 μm mode, and a coarse 300-600 μm tail attributed to Mg. SEM-EDS mapping evidences a mosaic microstructure: isometric Pd-rich domains (~10-60 μm), platy Ti-rich fragments (~20–80 μm), local Pd-Ti colocalization at interfaces (interpreted as a prereaction state for Pd-Ti intermetallics), and predominantly intergranular, dispersed Mg. The combined data indicate abundant high-energy interfaces and shortened diffusion paths created by mechanical activation, which are favorable for accelerating (de)hydrogenation while avoiding premature formation of bulk intermetallics. The results provide a microstructure-guided route to tune capacity, kinetics, and cycling stability via controlled milling parameters and targeted thermal schedules that promote beneficial Pd↔Ti interfacial reactions while limiting Mg segregation and oxidation.

Keywords: *Pd-Ti-Mg, mechanical activation, XRD; SEM-EDS, granulometry, interphase boundaries, hydrogen storage.*

Introduction

Contemporary research in hydrogen energy focuses on developing materials that provide efficient solid-state storage and transportation of hydrogen. Intermetallic and nanostructured systems attract attention due to their high capacity and stability in cyclic absorption/desorption processes [1-3]. In particular, alloys with rare-earth metals, titanium and aluminum compounds, as well as carbon nanomaterials, show encouraging results. Achieving the target characteristics requires a deep understanding of the structural-phase state and physicochemical properties of such materials, as well as optimization of synthesis and post-processing methods to improve specific capacity, (de)hydrogenation kinetics, and durability [4,5].

When choosing a material for hydrogen storage, the following key parameters are usually considered: operating temperatures and pressures, reaction rate, and sorption capacity [6-10]. In this regard, as noted above, the creation of new materials for hydrogen energy requires detailed investigation of physical and mechanical properties, as well as complex processes occurring both in the bulk of the material and on its surface. Although numerous experimental studies have been reported, the theoretical understanding of the influence of alloying additions on the hydrogen absorption process in alloys is still insufficiently developed [11-14]. In this context, palladium and its alloys have traditionally been considered benchmark materials due to their high hydrogen permeability and pronounced catalytic properties. Palladium possesses several exceptional properties that allow it to be integrated into various hydrogen technologies. Pd is considered a unique material with a strong affinity for hydrogen, owing to both its catalytic and hydrogen-absorbing properties [15], and it plays a key role in the hydrogen economy [16]. One of the promising intermetallic systems for applications in hydrogen energy is the ternary Pd-Ti-Mg system. Interest in these materials is driven by their unique hydrogen absorption properties, as well as the possibility of their use for hydrogen storage and in fuel cells. Extensive studies of Pd hydrides have been conducted in various areas of fundamental science and engineering. An important characteristic of palladium is the ability to form hydrides of composition PdH_x (where $0 < x < 1$), which makes it one of the prime candidates for the development of hydrogen storage devices. Unlike their bulk counterparts, nanostructured materials appear to exhibit faster charge and discharge kinetics, longer lifetimes, and size-tunable thermodynamics [17]. Also, Mg-based materials, owing to their high theoretical capacity (7.6 wt.% for MgH_2), are considered promising candidates for hydrogen storage [18]. At the same time, they require modifications of crystal chemistry and defect structure in order to accelerate mass transport and lower the decomposition temperature of the hydride. Additional advantages of magnesium hydride include the abundance and low cost of Mg, its non-toxicity, and its low atomic and molar mass [19]. Titanium is widely investigated as an effective catalytic additive to MgH_2-based systems

[20-26]. It is known that in multicomponent systems of the $Mg_xTi_yH_z$ type, improved thermodynamic and kinetic parameters of (de)hydrogenation are achieved compared to the individual hydrides of Mg and Ti: for a number of compositions, hydrogen release is observed at lower desorption temperatures from the solid phase [27,28]. In this case, titanium is a suitable element not only as an activator of molecular hydrogen dissociation on the surface, but also due to its low reactivity toward Pd and Mg even at elevated temperatures; it promotes the formation of beneficial interfaces without excessive formation of secondary intermetallics and can serve as a diffusion "bridge" for H atoms [29-31].

From a mechanistic point of view, in ternary Pd-Ti-Mg systems a key role is played by the engineering of interphase boundaries and defect states. Palladium ensures rapid surface dissociation of molecular hydrogen and the delivery of atomic H to interfaces; titanium, possessing low chemical activity toward Mg and Pd, prevents the formation of "dead-end" phases, while simultaneously acting as a trap for H and as nucleation centers for hydrides. Magnesium serves as the main bulk reservoir, where hydrogen diffusion is governed by dislocation density, grain size, the degree of microstructural heterogeneity, and the presence of elastic lattice strains. Mechanical activation creates a combination of fine comminution, high-angle misorientations, supersaturation of solid solutions, and metastable intermetallics; collectively, this reduces diffusion paths, increases the fraction of high-energy grain boundaries, and facilitates the transition states required for (de)hydrogenation [32]. For the binary Mg-Pd subsystem, the possibility of forming intermetallic phases (Mg_6Pd, $Mg_{0.9}Pd_{1.1}$., etc.) has been demonstrated, which substantially influence hydrogen-absorbing properties; stable phases are observed already at the stage of mechanical alloying according to XRD data [33]. The addition of Ti further lowers the energy barrier for hydrogen diffusion and accelerates hydride formation; Ti-containing additives in MgH_2 systems improve the kinetics and enhance cyclic stability. In the powdered ternary system, this manifests as a combination of: (i) catalytic Pd nodes for H_2 dissociation; (ii) Ti-enriched interfaces facilitating H transport and partially gettering oxygen; (iii) an Mg matrix as the main bulk reservoir, in which shortened diffusion paths and a high defect fraction accelerate (de)hydrogenation.

In this context, mechanical activation of Pd-Ti-Mg appears particularly attractive: it enables far-from-equilibrium states with fine dispersity (tens to hundreds of nanometers), a high defect fraction, and a developed interphase contact Pd↔Ti↔Mg at moderate processing costs and without prolonged high-temperature treatments. Moreover, fine tuning of the processing parameters (mill speed and energy, time, BPR, atmosphere, interval/pause milling) makes it possible to control the competition between fragmentation and cold welding, regulate the rate of formation of Mg-Pd intermetallics, and the distribution of Ti.

Based on the outlined premises, the aim of the present work is to investigate the structural–phase state and its evolution after mechanical activation of Pd-Ti-Mg system powders as a candidate material for solid-state hydrogen storage and transportation. The focus is on the role of Pd/Ti/Mg interphase boundaries, the formation of intermetallic constituents and their contribution to the thermodynamics and kinetics of (de)hydrogenation, as well as the potential for subsequent optimization of synthesis and processing regimes to achieve the required balance of capacity, rate characteristics, and long-term stability.

Materials and methods

The powder mixtures studied in this work were fabricated from elemental powders of Pd, Ti, and Mg with particle sizes <50 μm and a purity of 99%, obtained from Hebei Suoyi New Material Technology, Hebei Province, China. According to the manufacturer's technical datasheet, the initial powders had a particle size of less than 30 μm. The use of micron-sized tungsten carbide powders was due to the need to form a fine-grained structure capable of improving the strength characteristics of the resulting hard alloys.

For mechanical activation, ~9 g of the pre-prepared charge was loaded into a 45 mL stainless-steel vessel with stainless-steel milling balls (10 mm in diameter) at a ball-to-powder mass ratio of 10:1. Loading was carried out in an argon glove box VGB-3C to avoid oxidation. The vessel was sealed and mounted on a Pulverisette 7 Premium Line planetary micro mill, Fritsch GmbH, Germany. Grinding was performed at a rotation speed of 500 rpm. This speed, according to the literature, is optimal for efficient mechanical alloying without excessive cold welding or risk of ignition, providing a high-energy impact regime sufficient for particle fragmentation and alloying at a controlled temperature.

Surface morphology was studied using high-resolution scanning electron microscopy (SEM, Tescan Mira). The obtained micrographs enabled visualization of particle aggregation and shape characteristics. To determine the phase composition of the Pd-Ti-Mg powder mixtures after mechanochemical activation, X-ray phase analysis was performed using an XpERT-PRO Panalytics diffractometer with a (Cu) Kα radiation source (λ = 1.5406 Å) in the 2θ range of 20° to 90° at room temperature. The particle size distribution of the powders was measured using laser diffraction with an automated particle size analyzer (Analysette-22 NeXT Nano, Fritsch, Germany), based on the principles of measuring the intensity of scattered radiation induced by particles of various diameters. Measurements were carried out in a liquid dispersion medium with ultrasonic treatment to break up agglomerates and obtain a representative distribution.

Results and discussion

On the obtained diffraction pattern (CuK_α, 2θ≈30-90°), a set of reflections is recorded that can be confidently indexed to the elemental metal phases of the Pd-Ti-Mg system (Figure 1). The highest intensity is shown by the peak in the region of 2θ≈40° (narrow, with a high signal-to-background ratio), corresponding to the (111) reflection of fcc palladium (Fm-3m, a=3.887 Å). Additional palladium reflections are traced in the regions 2θ≈46-47° ((200)), 67-69° ((220)), 81-83° ((311)), 85-87° ((222)), which is consistent with the JCPDS reference cards for fcc-Pd. The intensity ratios of the Pd reflections are close to the tabulated values, and there are no signs of pronounced texture (preferred orientation).

Titanium is present in two modifications. First, a cubic Ti phase is identified (denoted as Ti_c), interpreted as a metastable fcc modification (Fm-3m, a=4.060 Å), the formation of which is possible as a result of high-energy mechanical activation/mechanical alloying. Its reflections overlap the regions 2θ≈41-42° ((200)), ≈70° ((220)) and ≈82° ((311)), partially superimposing on the Pd peaks and contributing to the observed shoulders/asymmetry of the profiles. Second, the hexagonal α-form of titanium is present (Mg-type structure, $P6_3/mmc$, a=2.950 Å, c=4.681 Å): characteristic reflections are recorded in the regions 2θ≈35-36° ((100)), 38-39° ((002)) and 40-41° ((101)), where they partially coincide with the strong (111) Pd. This overlap explains the slight asymmetry of the main peak at ~40° and the presence of weak satellite maxima.

Magnesium is identified as a hexagonal phase (Mg-type structure, $P6_3/mmc$, a=3.209 Å, c=5.211 Å). For it, the (100) reflection is expected in the region of 2θ≈32-33°, (002) ~34-35°, (101) ~36-37°, as well as higher-angle (102), (110), (103) and others. Within the working scanning range, weak Mg peaks are visible on the low-angle edge (starting at approximately 32-33°) and several low-intensity maxima in the interval 58-75°, which is consistent with a small fraction of magnesium and the comparatively low scattering coefficient for a light element.

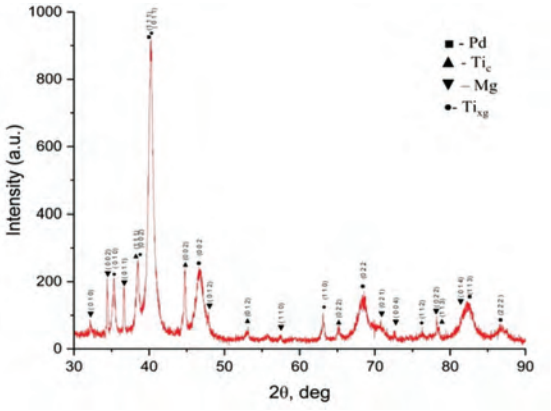

Figure 1. X-ray powder diffraction pattern of the Pd-Ti-Mg system

From the Q3(x) curve, the characteristic quantile diameters are determined: D10≈5 μm, D50≈20 μm, D90≈50 μm, which corresponds to a fairly wide, polydisperse system with span=(D90-D10)/D50≈2.25. The main contribution to the volume (≈70-80%) is formed by particles of the mid-size class 10-40 μm; the histogram in this zone is close to log-normal and shows a pronounced maximum around 15-25 μm. This interval is typical for the initial Pd and Ti powders and provides an acceptable compromise between bulk density and specific surface area. In the 0.8-5 μm region, a fine, low-intensity mode is recorded. Its origin may be associated with finely dispersed Pd/Ti fragments formed during sieving, transportation, or initial mechanical treatment, as well as with partial breakup of agglomerates in the analyzer's dispersing medium. The noted fine fraction increases the specific surface area and reactivity of the mixture; however, it can worsen rheology (caking, dusting) and accelerate oxidation of the active components. At the opposite end of the scale, a narrow coarse-grained mode of ~300-600 μm is observed (cumulative increment ≈5-10%). Taking into account particle morphology and density, it is reasonable to attribute it to the initial Mg powder. The presence of such a "tail" leads to bi-/multimodality of the mixture and potential segregation during storage and feeding (separation by fractions and density), and in mechanical activation processes to nonuniform transfer of impact energy: large Mg particles act as "hard inclusions," locally intensifying comminution, but at the same time worsening reproducibility and increasing the risk of cold welding.

The Q3(x) curve reaches a "plateau" around 50 μm (≈90-95% of the volume) and then makes a second step in the 300-600 μm zone, which is in good agreement with the visually multimodal histogram (Figure 2). There are practically no significant signs of oversized particles >1 mm; the upper tail is cut off near 1-1.5 mm.

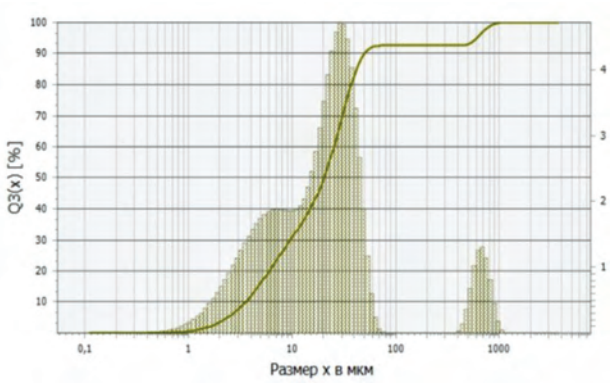

Figure 2. Particle size distribution of the Pd-Ti-Mg system

SEM-EDS mapping (Figure 3) demonstrates pronounced phase heterogeneity of the initial mixture, consistent with the results of X-ray phase analysis. Palladium forms compact isometric domains about 10-60

μm in size with smooth boundaries and limited intradomain variability of the Pd Lα1 intensity, which indicates a nearly single-phase nature of these areas. Titanium, in contrast, is represented by platy-scaly fragments of 20-80 μm, often forming packets with partial mutual overlap, which is typical of mechanically deformed hcp-Ti particles. Comparison of the Pd and Ti channels reveals regions of spatial overlap of signals, interpreted as zones of close interfacial contact. Given the thermodynamic stability of Pd-Ti intermetallics (PdTi, Pd2Ti, Pd3Ti), such colocalization can be considered a prereaction state, where the nucleation of ordered compounds is limited by small sizes and volume fraction and therefore does not manifest itself as separate lines in powder XRD.

The distribution of magnesium is qualitatively different. The Mg Kα1,2 intensity is low and fragmented; the signal is enhanced along intergranular regions and pores, which is consistent with the extremely low mutual solubility of Mg in Pd and Ti and the tendency toward interfacial segregation. The absence of large "pure" Mg inclusions in the field of view agrees with XRD, where magnesium appears as a minor phase with weak reflections. Analytical limitations should be taken into account: the low-energy Mg line is subject to absorption in the matrix and oxide films, therefore the quantitative contribution of Mg by EDS is likely underestimated; nevertheless, the topology of the distribution unambiguously indicates predominantly intergranular localization.

The morphological features visualized by the EDS maps are in good agreement with the granulometry (D10≈5 μm, D50≈20 μm, D90≈50 μm): the dominance of the 10-40 μm class corresponds to the volume fraction

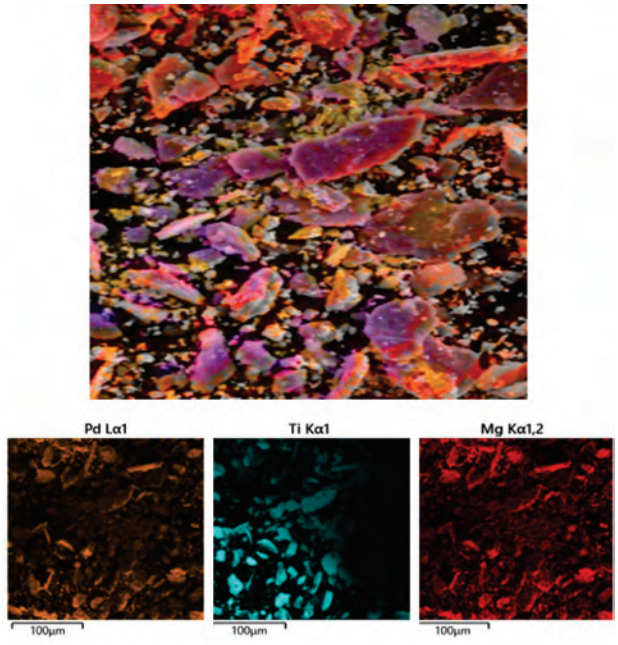

Figure 3. SEM-EDS maps of element distribution in Pd-Ti-Mg powder

of Pd/Ti particles, whereas finely dispersed fragments <5 μm form an intergranular "interlayer", increasing the Pd↔Ti contact area. Such an architecture favors solid-state interaction during subsequent thermomechanical treatment: coarsened domains provide stable heat supply, while the fine fraction provides short diffusion paths and an increased density of active boundaries.

Taken together, the observations confirm: (i) the presence of separated metallic phases Pd and Ti with stable interfacial contacts; (ii) dispersed intergranular localization of Mg; (iii) the absence of developed intermetallics at the bulk level, while maintaining a high probability of their nucleation at Pd↔Ti contact sites. These findings determine the strategy of subsequent processing-controlled heating/holding to intensify Pd-Ti diffusion while simultaneously limiting oxidation and segregation of Mg.

Conclusion

The conducted study shows that mechanical activation of the ternary Pd-Ti-Mg system forms a multicomponent microstructure in which the elemental phases fcc-Pd, α-Ti (hcp), and minor hcp-Mg with a small metastable fraction of fcc-Ti stably coexist. At the current processing stage, distinct reflections of intermetallics do not appear, which indicates either their extremely small volume fraction or submicron sizes of potential nuclei lying below the detection limit of powder XRD without profile analysis. At the same time, SEM–EDS mapping records local colocalization of Pd and Ti at contact areas, which can be interpreted as a prereaction state for the nucleation of ordered compounds such as PdTi/Pd_2Ti. This combination the absence of massive intermetallics in the presence of developed interphase boundaries is a favorable starting state for accelerated (de)hydrogenation.

Granulometry indicates a polydisperse, multimodal nature of the powder (D10≈5 μm, D50≈20 μm, D90≈50 μm) with a fine sub-5-μm mode and a coarse tail of ~300-600 μm attributed to Mg particles. This "architecture" of particles sets technological constraints and opportunities: the fine fraction increases specific surface area and defect density, shortens diffusion paths for H, and promotes rapid saturation; the medium class of 10-40 μm provides acceptable bulk density and packing reproducibility; the large Mg tail, by contrast, increases the risk of segregation and nonuniform energy transfer during milling. Consequently, controlled narrowing of the coarse tail (presieving/deagglomeration of Mg) and dosed retention of the fine fraction (with control of aggregation and oxidation) appear to be key for property stability and process scalability.

From the mechanistic standpoint, the body of data indicates that mechanical activation creates shortened diffusion paths, increases the fraction of high-energy boundaries, and forms a network of Pd↔Ti↔Mg interfaces. In this network, Pd acts as a catalyst for H_2

dissociation and as the "entry gates" for atomic hydrogen; Ti serves as a "diffusion bridge" and a potential nucleation center for hydride phases; Mg is the main bulk reservoir for hydrogen storage. The absence of massive intermetallics at the start helps avoid "dead-end" phases that block transport and preserves flexibility for subsequent fine tuning by heat treatment.

Overall, the results obtained confirm the feasibility of using mechanically activated Pd-Ti-Mg powders as a platform for solid-state hydrogen storage. The strategy for further optimization should rely on: (a) fine tuning of dispersity and the Pd↔Ti contact network with minimal loss of defectiveness; (b) controlled "turn-on" of intermetallic activity at interfaces without transition to bulk ordering; (c) engineering control of the surface chemistry of Mg to reduce mass-transfer barriers. Implementation of this strategy through controlled milling modes and thermocycling with in-situ diagnostics opens a pathway to materials with tunable capacity, accelerated (de)hydrogenation kinetics, and enhanced cycling stability, which is critical for practical applications in hydrogen energy.

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ИССЛЕДОВАНИЕ СТРУКТУРНО-ФАЗОВОГО СОСТОЯНИЯ МЕХАНОАКТИВИРОВАННОЙ СИСТЕМЫ PD-TI-MG

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В работе исследованы структурно-фазовое состояние и микроструктура тройных порошков Pd-Ti-Mg после механической активации как кандидатов для твёрдофазного хранения водорода. Исходные элементы подвергали высокоэнергетическому планетарному помолу (Pulverisette 7, 500 об/мин; BPR=10:1) в атмосфере аргона. По данным рентгенофазового анализа (CuKα) фиксируются фазы fcc-Pd (Fm-3m, a=3,887 Å), α-Ti (hcp, P63/mmc, a=2,950 Å, c=4,681 Å), метастабильная доля fcc-Ti (Fm-3m, a=4,060 Å) и минорный hcp-Mg (P63/mmc, a=3,209 Å, c=5,211 Å); отчётливые линии интерметаллидов в объёме не выявлены. Лазерная дифракция показала мультимодальное распределение размеров с D10≈5 мкм, D50≈20 мкм, D90≈50 мкм (span≈2,25), тонкой модой <5 мкм и грубозернистым «хвостом» 300-600 мкм, атрибутируемым Mg. SEM-EDS-картирование выявляет мозаичную микроструктуру: изометричные Pd-обогащённые домены (≈10-60 мкм), пластинчатые Ti-обогащённые фрагменты (≈20-80 мкм), локальную колокализацию Pd-Ti на интерфейсах (предреакционное состояние для Pd-Ti-интерметаллидов) и преимущественно межзеренную дисперсную локализацию Mg. Совокупность данных указывает на формирование укороченных диффузионных путей и высокоэнергетических границ, благоприятных для ускорения (де)гидрирования при отсутствии массивного образования интерметаллидов на текущей стадии. Показано, что управление параметрами помола позволяет целенаправленно усиливать полезные Pd↔Ti интерфейсные реакции при одновременном ограничении сегрегации и окисления Mg, настраивая ёмкостно-кинетические характеристики и циклическую стабильность.

Ключевые слова: *Pd-Ti-Mg; mechanical activation; XRD; SEM-EDS; granulometry; interphase boundaries; hydrogen storage.*

МЕХАНИКАЛЫҚ АКТИВАЦИЯЛАНҒАН PD-TI-MG ЖҮЙЕСІНІҢ ҚҰРЫЛЫМДЫҚ-ФАЗАЛЫҚ КҮЙІН ЗЕРТТЕУ

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Бұл жұмыста сутекті қатты күйде сақтауға арналған үміткер материал ретінде механикалық активтендірілген үштік Pd-Ti-Mg ұнтақтарының құрылымдық-фазалық күйі мен микроқұрылымы зерттелді. Бастапқы элементтік ұнтақтар аргон атмосферасында жоғарыэнергиялы планетарлық диірменде өңделді (Pulverisette 7, 500 айн/мин; BPR=10:1). Рентгенфазалық талдау (CuKα) fcc-Pd (Fm-3m, a=3.887 Å), α-Ti (hcp, P63/mmc, a=2.950 Å, c=4.681 Å), метатұрақты fcc-Ti (Fm-3m, a=4.060 Å) және аз мөлшердегі hcp-Mg ($P6_3/mmc$, a=3.209 Å, c=5.211 Å) фазаларын көрсетті; көлемде айқын интерметаллидтік шыңдар байқалмады. Лазерлік дифракция бөлшек өлшемдерінің мультимодальды таралуын анықтады: D10≈5 мкм, D50≈20 мкм, D90≈50 мкм (span≈2.25), <5 мкм майда мода және 300-600 мкм ірі «құйрық» (Mg-ге тән). SEM–EDS карталау мозаикалық микроструктураны айқындады: Pd-ға бай изометриялық домендер (≈10-60 мкм), Ti-ға бай жапырақша-пластиналы фрагменттер (≈20-80 мкм), интерфейстерде Pd-Ti колокализациясы (Pd-Ti интерметаллидтерінің алдындағы реакцияға дейінгі күй) және негізінен түйір аралық аймақтарда дисперсті Mg. Жиынтық деректер механикалық активтендіру нәтижесінде диффузиялық жолдардың қысқаратынын және жоғарыэнергиялы шекаралар үлесінің артатынын көрсетеді, бұл (де)сутектену кинетикасын жеделдетуге қолайлы, әрі көлемдік интерметаллидтердің ерте түзілуін болдырмайды. Процестік параметрлерді дәл баптау Pd↔Ti интерфейстік реакцияларын күшейтіп, Mg-дің сегрегациясы мен тотығуын шектей отырып, сыйымдылық-кинетикалық сипаттамаларды және циклдік тұрақтылықты оңтайландыруға мүмкіндік береді.

Түйін сөздер: *Pd-Ti-Mg, механикалық активтендіру, РФА, SEM-EDS, гранулометрия, фазааралық шекаралар, сутекті сақтау.*