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KINETICS AND TEMPERATURE DEPENDENCES OF SHAPE CHANGES OF PALLADIUM CANTILEVERS DURING DEGASSING OF α-PdH_n ALLOYS

O.M. Liubymenko
Donetsk National Technical University, Drohobych, Lviv region, Ukraine
E-mail: e.n.lyubimenko@gmail.com

Abstract. The kinetics and temperature dependences of deformation of α-PdH_n alloy palladium cantilevers during degassing at 240…350 °C were investigated. The maximum reverse bending depends on the initial hydrogen content, with the largest deformation observed after saturation at the highest studied hydrogen pressure. Multi-stage degassing revealed a non-monotonic bending behavior caused by hydrogen concentration profile redistribution and stress relaxation. A mechanism of reverse deformation based on concentration-dilation gradient formation in transient α-PdH_n alloys is proposed.

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INTRODUCTION

The study of the processes of interaction of metals with hydrogen is one of the urgent problems of modern solid-state physics and materials science. Of particular interest among metal–hydrogen systems is the Pd–H system [1–4], which is due to the unique ability of palladium to reversibly absorb significant amounts of hydrogen, as well as its widespread use in hydrogen energy, membrane technologies for hydrogen purification, sensor systems, and catalysis [5, 6]. Due to its well-studied physicochemical properties and the simplicity of the phase diagram, the Pd–H system is a classic model for studying the fundamental laws of hydrogen diffusion in metals [5–9].

Palladium retains a face-centered cubic (fcc) crystal structure over a wide temperature range. During interaction with hydrogen, its atoms occupy predominantly octahedral interstitial positions of the crystal lattice, since they are the most energetically favorable [5, 9–12]. The filling of these positions is accompanied by a local expansion of the crystal lattice, which arises as a result of the interaction of the electron shells of hydrogen and palladium atoms [13]. The combination of such local dilations leads to an increase in the lattice parameter and, in the presence of an inhomogeneous distribution of hydrogen, forms concentration-dependent deformations and internal mechanical stresses in the α-PdH_n alloy [12, 14].

The kinetics of the formation and relaxation of this stress-strain state is determined by the diffusion mobility of hydrogen. It is known that the transfer of hydrogen in palladium is satisfactorily described by the jump mechanism of diffusion, confirmed by the results of quasi-elastic neutron scattering [5, 11, 13]. With increasing temperature, the hydrogen diffusion coefficient increases [6, 15], the leveling of the concentration profile accelerates, and the relaxation rate of elastic deformations changes accordingly.

An important factor determining the mechanical response of the Pd–H system is the temperature dependence of the concentration dilation [5, 6, 15–17]. At room temperature, the relative difference in specific volumes of the α- and β-phases is about $\Delta V/V = 10.59\%$, but with increasing temperature it monotonically decreases and practically disappears near the critical phase transition temperature (Tcr ≈ 292 °C) [4]. This indicates a significant decrease in the volume effect of hydrogen dissolution with temperature and gives reason to expect a corresponding change in the amplitude of deformations during palladium degassing. Despite the significant amount of research into the processes of hydrogen absorption by palladium, the regularities of degassing [18, 19], especially their connection with the development of concentration gradients, internal stresses and deformation of thin palladium elements [20–23], have not been studied sufficiently. This is especially true for the temperature dependences of the kinetics of deformation of palladium cantilevers during hydrogen release from α-PdH_n alloys. The study of these processes is important both for the development of the physics of metal-hydrogen systems and for the creation of highly sensitive hydrogen sensors, microelectromechanical systems and functional elements [24, 25], the operation of which is based on hydrogen-induced deformations. The aim of the work is to study the kinetics and temperature patterns of deformation of palladium cantilevers during degassing of α-PdH_n alloys, as well as to establish the relationship between the rate of hydrogen release, the experimental temperature and the magnitude of the deformation response of the cantilever.

METHODS AND APPARATUS

Experimental studies were performed using an improved hydrogen-vacuum unit (HVU), the design and principle of which are described in detail in [26]. Palladium cantilevers with a purity of 99.9% and dimensions of 68×5.5×0.27 mm were used for the study. To ensure one-sided penetration and exit of hydrogen, a solid copper coating 750 nm thick, which is impermeable to hydrogen, was applied to one of the surfaces of each sample by electrolytic deposition [27].

After installing the samples in the working chamber of the hydrogen-vacuum unit, they were heated to the specified temperature of the study. To stabilize the temperature field and relax thermal stresses, the samples were kept in isothermal conditions for 30 min. After reaching thermal equilibrium, hydrogen was supplied to the working chamber to a pressure of 0.27 MPa, ensuring

palladium saturation to concentrations corresponding to the formation of the α-PdH_n alloy in the α-phase of the Pd–H system. After the saturation process was completed, an isothermal hold was performed, necessary for the relaxation of concentration-induced stresses and the formation of a uniform concentration distribution of hydrogen in the volume of the material. Further study of the kinetics of the deformation response was carried out under conditions of unilateral degassing of the α-PdH_n alloy by stepwise reduction of the hydrogen pressure by ΔP_{H_2} = 0.15; 0.09, and 0.03 MPa. Changes in the geometric shape of palladium cantilevers made of the α-PdHn alloy were continuously recorded using a cathetometer and a video fixation system with subsequent analysis of the kinetics of shape change.

EXPERIMENT

Experimental studies were performed at temperatures of 240, 280, and 320 °C (Figs. 1–3). The results of similar studies conducted at a temperature of 350 °C are presented and analyzed in detail in [21].

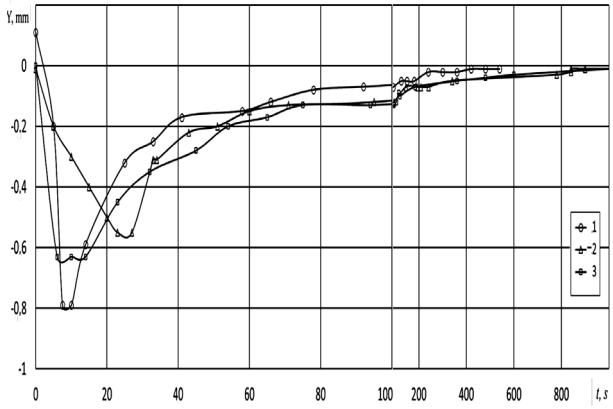


Fig. 1. Time dependences of the shape change of the cantilever at a temperature of 320 °C in the process of one-sided degassing when the pressure changes by ΔP_{H_2}: 1 – 0.15 MPa; 2 – 0.09 MPa; 3 – 0.03 MPa

Within the framework of a series of experiments conducted at a temperature of 320 °C, an α-PdH_n alloy was obtained and successive further degassing was carried out in order to study the kinetics of cantilever deformation, which is accompanied by a change in the hydrogen content in the material (see Fig. 1). During degassing at the first stage, the pressure was reduced from 0.27 to 0.12 MPa, i.e. by ΔP_{H_2} = 0.15 MPa, for 7.56 s at a rate of 0.0198 MPa/s. The minimum bending of the cantilever was -0.79 mm and was recorded 8 s after the start of degassing. The occurrence of negative bending indicates the formation of a reverse hydrogen concentration gradient, when the intensive release of hydrogen from the surface layers changes the distribution of internal stresses in the material. During further exposure, the deformation gradually decreased, and after 8 min it was only -0.01 mm, i.e. about 1.27% of the initial negative bending. During the next 2 min, no noticeable changes were observed, which indicates an almost complete extinction of the reverse deformation. At the second stage of degassing, the pressure was reduced from 0.12 to 0.03 MPa, the increase in pressure change was ΔP_{H_2} = 0.09 MPa. The process lasted 16.06 s at a pressure decrease rate of 0.0056 MPa/s. The maximum reverse bending was -0.55 mm and was achieved 23 s after the start of degassing. Compared with the first stage of degassing, a decrease in the absolute value of the negative bending and an increase in the time to achieve it were observed. This indicates a slower formation of the concentration gradient at a lower rate of pressure change. During further holding, the deflection decreased to zero, and subsequently a slight positive deviation of +0.01 mm was observed after 14.38 min. Further holding for 3 min did not lead to noticeable changes in the position of the cantilever.

At the third stage of degassing, the pressure was reduced from 0.03 MPa to 1.33 kPa, i.e. by ΔP_{H_2} = 0.03 MPa, for 6.48 s at a rate of 0.00463 MPa/s. The maximum negative deflection was -0.63 mm and was recorded 9.04 s after the start of degassing. Over the next 18 min, there was a gradual decrease in deformation to zero. An additional holding for 2 min did not change the position of the sample, which indicates the completion of the main stage of degassing and relaxation of internal stresses.

Analysis of the degassing results showed that the release of hydrogen from palladium is accompanied by the occurrence of reverse bending of the cantilever. Its amplitude and kinetic characteristics are determined by the initial hydrogen concentration in the material, the magnitude of the pressure drop, the rate of its decrease, as well as the features of the concentration field formed at the stage of preliminary one-sided saturation.

The maximum negative value of the bending (-0.79 mm) was recorded during the first stage of degassing after saturation of the α-PdH_n alloy. to a pressure of 0.27 MPa. The result obtained indicates that with an increase in the hydrogen content in palladium, the concentration gradient that occurs at the initial stage of degassing increases, which, in turn, leads to the development of greater internal hydrogen stresses and, accordingly, a more pronounced deformation response of the cantilever.

Experimental studies performed at a temperature of 320 °C confirmed the staged nature of the shape change of the cantilever from the α-PdH_n alloy during degassing. At the initial stage, a rapid formation of reverse bending is observed, which persists for several seconds. Subsequently, as the hydrogen diffusion redistribution occurs, the cantilever gradually straightens, due to the relaxation of concentration-induced stresses, the alignment of the hydrogen distribution in the α-phase of the Pd–H system, and the establishment of a new equilibrium position. Thus, the results of the study showed that the deformation response of the palladium cantilever at a temperature of 320 °C is determined by the interaction of the processes of hydrogen diffusion transport, the formation of a temporary concentration gradient, the development of internal hydrogen-concentration stresses, and their subsequent relaxation. The combination of these processes determines the kinetics of reverse bending and subsequent straightening of the cantilever during degassing of the α-PdH_n alloy.

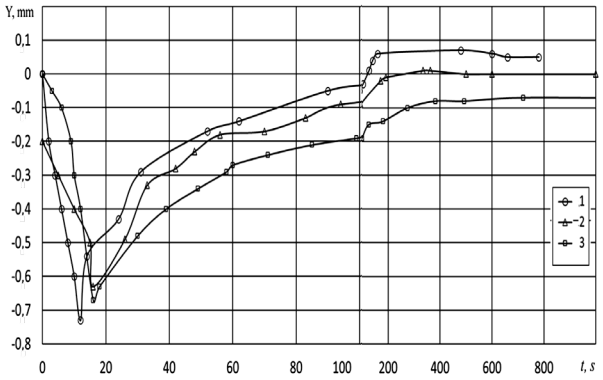


Fig. 2. Time dependences of the cantilever deformation at a temperature of 280 °C during unilateral degassing with pressure change by ΔP_{H_2}: 1 – 0.15 MPa; 2 – 0.09 MPa; 3 – 0.03 MPa

To confirm the results obtained, a similar series of experiments was performed at a temperature of 280 °C (Fig. 2). After completion of unilateral hydrogen saturation at a temperature of 280 °C, a sequential stepwise pressure reduction was performed with subsequent vacuum holding after each stage.

At the first stage, the pressure was reduced from 0.27 to 0.12 MPa over a period of 5.44 s, which corresponded to a rate of 0.0275 MPa/s. The maximum reverse bending of the cantilever was -0.73 mm and was achieved 14 s after the start of degassing. Its occurrence is associated with the formation of a reverse hydrogen concentration gradient, which is formed as a result of a more intense release of hydrogen from the surface layers compared to the inner layers of the alloy. This leads to a redistribution of concentration-induced stresses and the appearance of deformation in the opposite direction. During further vacuum holding, the bending gradually decreased, the cantilever returned to its original position, after which a slight positive deviation (+0.07 mm) was established, which remained practically unchanged for the next 3 min. At the second stage of degassing, the pressure was reduced from 0.12 to 0.03 MPa in 10.80 s, which corresponded to a speed of 0.0083 MPa/s. The maximum reverse bending was -0.63 mm and was observed 16 s after the start of the process. During further vacuum holding, the deformation amplitude gradually decreased, and after approximately 6.02 min the cantilever reached its original position. Further holding in vacuum for 4 min was not accompanied by noticeable changes in deformation.

In the third stage, the pressure was reduced from 0.03 MPa to 1.33 kPa over 5.49 s at a rate of 0.00546 MPa/s. The maximum reverse bending was -0.67 mm and was recorded 16 s after the start of degassing. Further, during the vacuum holding, the cantilever gradually straightened. After 22 min, the residual deformation was -0.05 mm, which corresponds to approximately 7.4% of the maximum amplitude of reverse bending. An additional five-minute holding did not ensure complete relaxation of the residual deformation.

Thus, the results of the studies showed that at a temperature of 280 °C, unilateral saturation of palladium with hydrogen is accompanied by the rapid formation of positive bending of the cantilever, its subsequent partial straightening and establishment of a residual equilibrium position. With increasing hydrogen pressure, the magnitude of the residual positive bending increases from +0.11 to +0.25 mm, which indicates an increase in concentration-induced deformations and incomplete relaxation of internal stresses after the saturation process is completed. During degassing, on the contrary, a negative bending is formed, the amplitude of which is determined by the initial hydrogen content in palladium, the magnitude of the pressure change and the features of the diffusion redistribution of hydrogen in the alloy volume. The obtained results confirm that the deformation response of the palladium cantilever at 280 °C is determined by the combined action of hydrogen diffusion processes, the formation of a temporary concentration gradient, the emergence of concentration stresses and their subsequent relaxation. The established patterns became the basis for further studies of deformation processes at a lower temperature 240 °C, since it was in this temperature range that the largest values of the maximum cantilever bending were recorded during saturation.

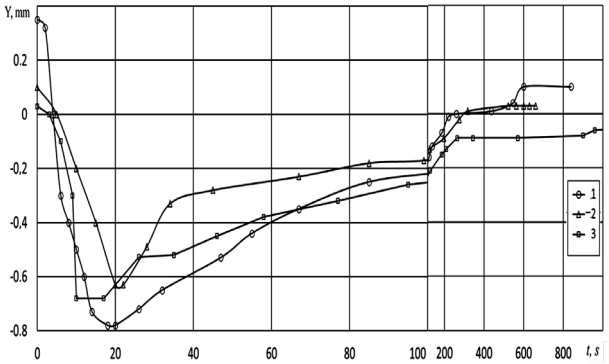


Fig. 3. Time dependences of the shape change of the cantilever at a temperature of 240 °C in the process of one-sided degassing with a change in pressure by ΔP_{H_2}: 1 – 0.15 MPa; 2 – 0.09 MPa; 3 – 0.03 MPa

At a temperature of 240 °C (Fig. 3) during degassing at the first stage, the pressure in the chamber was reduced from 0.27 to 0.12 MPa (ΔP_{H_2} = 0.15 MPa) for 8.55 s, which corresponded to a speed of 0.0175 MPa/s. 10 s after the start of degassing, the cantilever reached its maximum reverse bending, which was -0.78 mm. Its occurrence is associated with the formation of a reverse hydrogen concentration gradient, which arises as a result of a more intense release of hydrogen from the near-surface layers of palladium. During the subsequent vacuum exposure, the deformation gradually disappeared and the cantilever reached the initial position, and after 450 s a slight positive deviation (+0.10 mm) was observed. The next 6-minute exposure was not accompanied by further changes in the deformation.

At the second stage of degassing, the pressure was reduced from 0.12 to 0.03 MPa (ΔP_{H_2} = 0.09 MPa) for 7 s. The maximum reverse bending was -0.68 mm and was recorded 11 s after the start of degassing. During the vacuum exposure, the bending value gradually decreased: first the cantilever returned to the zero position, and after approximately 6.28 min a slight positive deviation (+0.03 mm) was established. Further

exposure in vacuum for 5 min did not cause noticeable changes in the deformation state.

At the third stage of degassing, the pressure was reduced from 0.03 MPa to 1.33 kPa (ΔP_{H_2} = 0.03 MPa) for 5 s, which corresponded to a rate of 0.006 MPa/s. The maximum reverse bending (-0.68 mm) was observed 10 s after the start of the process. Further, during the vacuum holding, the cantilever gradually straightened. After 25 min, the residual deformation was -0.05 mm, which corresponds to 7.35% of the maximum bending amplitude. An additional 5-minute holding did not ensure complete relaxation of the residual deformation. The results obtained indicate that at a temperature of 240 °C, the deformation of the palladium cantilever, as at higher temperatures, has a pronounced staged nature. At the degassing stage, a reverse bend is formed, the amplitude of which is determined by the initial hydrogen concentration, the rate of pressure decrease, and the features of the diffusion redistribution of hydrogen in the material. Further straightening of the cantilever is due to the relaxation of internal stresses and the gradual alignment of the hydrogen concentration field in the α-phase of the Pd–H system.

RESULTS AND DISCUSSION

Comparison of the results of experiments performed at temperatures of 240, 280, 320, and 350 °C shows that the deformation of a palladium cantilever made of α-PdH_n alloy during degassing with a sequential decrease in hydrogen pressure by ΔP_{H_2} = 0.15; 0.09, and 0.03 MPa is characterized by common kinetic patterns (Fig. 4). In all studied cases, hydrogen desorption is accompanied by the formation of a negative bending of the cantilever, which is a consequence of the occurrence of a reverse hydrogen concentration gradient along the thickness of the sample and the associated redistribution of concentration stresses.

At the first stage of degassing, which corresponds to the largest decrease in hydrogen pressure (ΔP_{H_2} = 0.15 MPa), the maximum negative bends in absolute value are observed. At the same time, the temperature dependence of the bend amplitude is non-monotonic: the values obtained at 320 and 350 °C occupy an intermediate position between the results for 240 and 280 °C. This feature indicates a complex relationship between the rate of hydrogen desorption, its diffusion transfer in palladium and the relaxation of concentration stresses. With a further decrease in pressure to ΔP_{H_2} = 0.09 MPa, a minimum amplitude of negative bend is observed for all temperatures. At the same time, with a decrease in pressure to ΔP_{H_2} = 0.03 MPa, the magnitude of the reverse bend increases again, although it remains smaller than at the first stage of degassing. This non-monotonicity indicates that the process of hydrogen removal is determined not only by its average content in palladium, but also by the evolution of the concentration profile in the volume of the material.

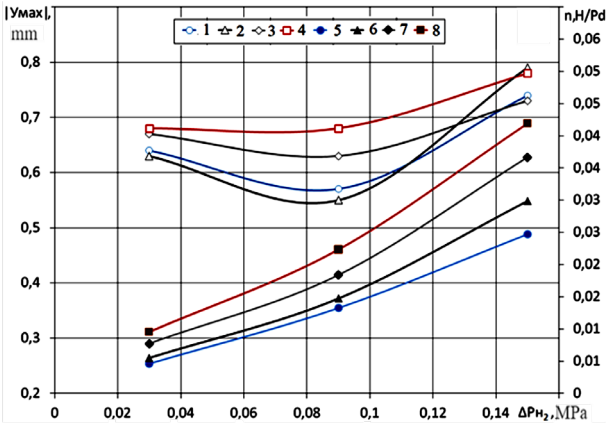


Fig. 4. Dependence of the maximum reverse bending of the cantilever (1–4) and the change in hydrogen concentration n (H/Pd) (5–8) on the difference in hydrogen partial pressure ΔP(H_2) at different experimental temperatures during the degassing process: 1, 5 – 240 °C; 2, 6 – 280 °C; 3, 7 – 320 °C; 4, 8 – 350 °C

The amplitude of the maximum deformation during degassing in absolute values is smaller than during hydrogen saturation. This is explained by the difference in the physical mechanisms of both processes. During saturation, the determining factor is the concentration dilation of the crystal lattice due to hydrogen penetration, while during degassing, its exit from palladium dominates, which is accompanied by a gradual decrease in local deformations and relaxation of internal stresses [28].

The largest negative curves in absolute value are observed after preliminary saturation of palladium to the maximum hydrogen pressure studied. This indicates that the initial hydrogen content determines the magnitude of the concentration gradient that is formed during degassing, and therefore the amplitude of the reverse deformation. After a long exposure in vacuum, the deformation of the cantilever is significantly reduced, but in some experiments a slight residual bend remains, indicating incomplete relaxation of concentration stresses.

To explain the established patterns, a model of a temporary gradient alloy α-PdH_n is proposed. After reducing the hydrogen pressure, the surface layers of the cantilever are the first to lose hydrogen, while in the inner layers its concentration still remains elevated. As a result, a reverse hydrogen concentration gradient is formed, which is accompanied by inhomogeneous concentration dilation of the crystal lattice.

The temporary gradient state of α-PdH_n can be conditionally considered as a set of layers with different hydrogen contents. Due to the mechanical connection between them, their free reduction is impossible, which leads to the incompatibility of local deformations and the appearance of internal stresses. By its mechanical nature, this process is similar to the operation of a bimetallic cantilever, but is determined not by the difference in thermal expansion coefficients, but by the concentration dilation gradient formed by the inhomogeneous distribution of hydrogen.

As further degassing proceeds, the concentration profile gradually levels off, the difference in local deformations between individual layers decreases, and with it the level of internal stresses decreases. This leads to a gradual straightening of the cantilever. At the initial stage of degassing, when the concentration gradient is maximum, the largest negative bend is formed. Later, as hydrogen is redistributed and stresses relax, the magnitude of the bend changes non-monotonically.

At a constant temperature, the hydrogen diffusion coefficient in palladium can be considered constant within the accepted approximation, therefore, the kinetics of degassing deformation is determined mainly by the initial saturation level of the material, the magnitude of the change in hydrogen pressure, and the rate of diffusion transfer of hydrogen from the internal regions to the surface. The higher the initial hydrogen concentration, the greater the concentration gradient formed at the initial stage of degassing and the greater the amplitude of negative bending.

Thus, the deformation of a palladium cantilever during degassing is determined by the interaction of the processes of hydrogen desorption, its diffusion transfer in the palladium volume, and the relaxation of concentration stresses. Negative bending is a mechanical manifestation of the uneven release of hydrogen from the temporary gradient alloy α-PdH_n and reflects the kinetics of restoring the equilibrium state of the Pd–H system. The results obtained deepen the understanding of the mechanisms of palladium degassing and can be used in the creation of palladium sensitive elements of hydrogen sensors, in which the magnitude and direction of the cantilever bending are informative parameters for controlling hydrogen desorption processes.

CONCLUSIONS

1. It was experimentally established that in the temperature range of 240…350 °C, the degassing of a palladium cantilever from the α-PdH_n alloy is accompanied by the formation of negative bending, which occurs as a result of the formation of a reverse hydrogen concentration gradient along the thickness of the sample and the relaxation of concentration stresses.

2. It was established that the amplitude of the maximum negative bending is determined by the initial degree of palladium saturation with hydrogen. The largest deformations in absolute value are observed after preliminary saturation to the maximum studied hydrogen pressure, while with a decrease in the initial hydrogen concentration, the bending amplitude decreases.

3. It is shown that during the three-stage degassing of the α-PdH_n alloy (ΔP_{H_2} = 0.15; 0.09, and 0.03 MPa) the dependence of the maximum negative bending on the degree of degassing is non-monotonic. The largest bend is formed at the initial stage of degassing, after which its magnitude decreases due to the diffusion redistribution of hydrogen and relaxation of internal stresses.

4. It was established that the mechanism of reverse deformation of the palladium cantilever during degassing is similar to the mechanism of deformation of bimetallic elements. Unlike bimetals, where the bend is determined by the difference in thermal expansion coefficients, in the α-PdH_n alloy it is due to the concentration dilation of layers with different hydrogen contents, which leads to the emergence of internal concentration stresses.

5. It is shown that after the completion of degassing, in some cases, a residual bend of the cantilever remains, the magnitude of which depends on the initial degree of saturation and the conditions of hydrogen removal. This indicates an incomplete relaxation of concentration stresses after the completion of the degassing process.

6. It was established that the kinetics of the reverse deformation is determined by the interaction of the processes of hydrogen desorption from the surface, its diffusion transfer in the volume of palladium, reorganization of the concentration profile and relaxation of internal stresses. At a constant temperature, the initial hydrogen content in palladium, the magnitude of the change in hydrogen pressure and the rate of diffusion redistribution of hydrogen have a decisive influence on the amplitude and kinetics of bending.

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КІНЕТИКА ТА ТЕМПЕРАТУРНІ ЗАЛЕЖНОСТІ ФОРМОЗМІНЕННЯ ПАЛАДІЄВИХ КАНТИЛЕВЕРІВ ПІД ЧАС ДЕГАЗАЦІЇ СПЛАВІВ α-PdH_n

О.М. Любименко

Досліджено кінетику та температурні залежності формозмінення паладієвих кантилеверів зі сплаву α-PdH_n під час дегазації в температурному інтервалі 240…350 °C. Встановлено, що величина максимального зворотного вигину визначається початковим вмістом водню у сплаві: найбільші негативні деформації спостерігаються після попереднього насичення до максимального дослідженого тиску водню. Показано, що під час багатостадійної дегазації залежність максимального вигину від ступеня дегазації має немонотонний характер, що зумовлено перебудовою концентраційного профілю водню, релаксацією та перерозподілом концентраційних напружень. Запропоновано механізм дегазаційного формозмінення, згідно з яким зворотний вигин кантилевера визначається формуванням градієнта концентраційної дилатації в тимчасовому градієнтному сплаві α-PdH_n.