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STRUCTURE, PHASE TRANSFORMATIONS,  
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## Effect of Temperature on the Form Change of a Palladium Plate during Its One-Sided Saturation with Hydrogen

M. V. Gol'tsova and E. N. Lyubimenko

Donetsk National Technical University, ul. Artema 58, Donetsk, 83001 Ukraine

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**Abstract**—Effect of temperature on the regularities and scale of form changes of a palladium plate has been studied in a cantilever mode of fixation of a specimen, during its one-sided saturation with hydrogen. The form change of the plate is confirmed to be developed in two stages. During the first short-time stage, the maximum form change (bending) of the plate is reached. The temperature dependence of the maximum bend of the plate is described by an extremal function. During the second stage, which is of substantially longer duration, a plate straightening takes place. At low temperatures, the residual steady-state bend of the plate is 20–30% of the maximum form change. As the temperature increases, the bend reversibility increases and at 320–350°C the phenomenon becomes completely reversible. The mechanism of form change is discussed, and its phenomenological analysis is given.

**Keywords:** hydrogen, palladium plate, gradient alloy, concentration stresses, form change

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### INTRODUCTION

Earlier, we have studied, in a cantilever mode of fixation of a specimen, the form change (bending) of a palladium plate as a function of gaseous hydrogen pressure at a constant temperature (240°C). It was shown [1] that, during one-sided saturation of a plate with hydrogen under conditions corresponding to the  $\alpha$ -Pd solid solution in the Pd–H phase diagram, the plate undergoes a two-stage bending.

During the first stage, the bending of the plate develops quite quickly and the maximum deflection is reached in 10–20 s. In this case, the increase in the hydrogen pressure causes a substantial increase in the maximum bending of the plate; at  $P_{H_2} = 0.43$  MPa, the deflection reaches 10 mm. During the second stage of hydrogen saturation, the plate straightens slowly. In this case, either almost complete reversibility of bending is reached or a slight residual (steady) bend takes place.

The present study is aimed at the investigation of the effect of temperature on the regularities and amounts of form changes of a palladium plate during its one-sided saturation with hydrogen.

### EXPERIMENTAL

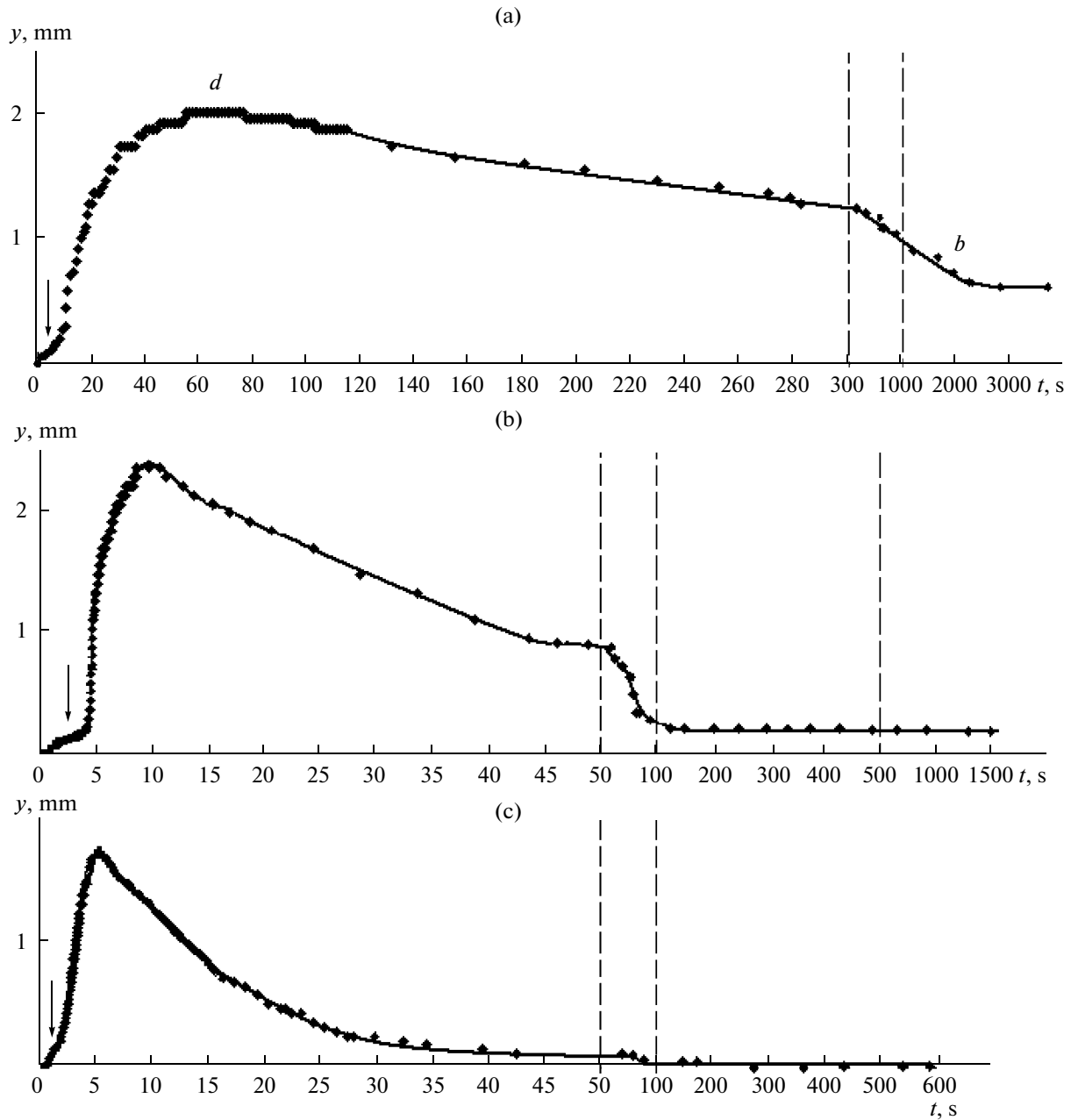
The sample under study has been prepared in the form of a thin pure palladium plate (68 × 5.5 × 0.27 mm in size). The sample has been annealed at 700°C for 60 min (and subsequently cooled in the furnace). One side of the plate has been electroplated with copper.

The investigations were performed in a VVU-4 vacuum–hydrogen setup, which allowed us to study the form change of the palladium plate at temperatures to 360°C at hydrogen pressures from 0.01 to 2.5 MPa. The setup consists of a working chamber and four service units; it has been described in detail in [1].

When preparing the experiment, the overhung end of the sample was mounted so that the copper-plated surface of the plate was directed upward. After mounting, the working part of the sample was equal to 63 mm. To relax residual stresses, the fixed sample was subjected to a low-temperature annealing directly in the working chamber; namely, the sample was slowly heated to 240°C at a rate of 3 K/min and subsequently cooled with the furnace. After three cycles of such annealing, the sample usually becomes insensitive to subsequent heatings and coolings (no noticeable bending is observed); thus, the sample is ready for the experiments.

The experiments were performed as follows. The sample was slowly heated (at a rate of 3 K/min) to a desired temperature and held at this temperature for 20 min. After that, diffusionally purified hydrogen was fed into the working chamber to a given pressure. The variations in the bending of the plate were recorded from the very onset of hydrogen feeding ( $t = 0$  s) using a quartz window, a cathetometer, and a video camera (Samsung).

The measurements were always realized under isothermal conditions. The temperature was maintained and measured accurate to  $\pm 1$  K using a TRTs-02-plus



**Fig. 1.** Time dependences of the form change of a palladium plate at  $P_{H_2} = 0.03$  MPa and temperatures (a) 110, (b) 240, and (c) 350°C.

instrument. After recording, the video tape was analyzed frame-by-frame using a Sony Vegas program; this allowed us to obtain the time dependence of the sample bending with the frame accuracy of 0.04 s. The accuracy of the measurement of the initial position of the free end of the plate is  $\pm 0.03$  mm. The use of video recording is of particular importance for the first seconds of experiments, when the form change of the sample develops rather quickly. In accordance with the assigned task, the hydrogen-induced form change of the palladium plate was studied using the above method in a temperature range of 110–350°C.

## RESULTS AND DISCUSSION

The first experiments were performed at a pressure  $P_{H_2} = 0.03$  MPa at temperatures of 110, 240, and 350°C. The obtained time dependences of the form change of the palladium plate are given in Fig. 1.

It follows from Fig. 1 that, in accordance with [1], the form change of the plate is realized in two time stages. At the first stage, there occurs a rapid bending of the plate; at all the temperatures under study, the plate begins to flex immediately after the onset of the hydrogen supply to the working chamber. In all the

**Table 1.** Effect of temperature on the form change of a palladium plate at a hydrogen pressure  $P_{H_2} = 0.03$  MPa

No	$T, ^\circ\text{C}$	$\Delta t_{\text{onset}}, \text{s}$	$\Delta t_{\text{max}}, \text{s}$	$y_{\text{max}}, \text{mm}$	$\Delta t_{\text{st}}, \text{s}$	$y_{\text{st}}, \text{mm}$	$\frac{y_{\text{st}}}{y_{\text{max}}} \times 100\%$	$n, [\text{Pd}/\text{H}]$	$D, \text{m}^2/\text{s}$
	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>
<b>1</b>	110	1.35	55	1.94	2700	0.618	31.9	0.0184	$2.71 \times 10^{-10}$
<b>2</b>	130	1.0	77	1.89	2400	0.40	21.4	0.0172	$3.95 \times 10^{-10}$
<b>3</b>	150	2.56	30	1.99	2400	0.17	8.9	0.0160	$5.55 \times 10^{-10}$
<b>4</b>	170	1.57	16	2.06	600	0.03	1.5	0.0146	$7.56 \times 10^{-10}$
<b>5</b>	200	1.35	18.84	2.40	489	0.218	9.1	0.0129	$1.14 \times 10^{-9}$
<b>6</b>	220	2.20	17.4	2.47	160	0.22	8.8	0.0117	$1.47 \times 10^{-9}$
<b>7</b>	240	2.61	11	2.33	121	0.218	9.3	0.0105	$1.85 \times 10^{-9}$
<b>8</b>	260	1.3	8.92	2.22	133	0.10	4.7	0.0097	$2.28 \times 10^{-9}$
<b>9</b>	280	0.94	7.4	2.22	540	0.10	4.7	0.0090	$2.78 \times 10^{-9}$
<b>10</b>	300	3.1	11.84	1.84	1200	0.07	3.8	0.0081	$3.33 \times 10^{-9}$
<b>11</b>	320	1.08	8.12	1.96	900	0	0	0.0074	$3.95 \times 10^{-9}$
<b>12</b>	350	1.12	4.69	1.75	277	0	0	0.0062	$5.0 \times 10^{-9}$

cases, at the moment of reaching the given pressure  $P_{H_2} = 0.03$  MPa (shown by arrows in Fig. 1), the plate deflection can already be easily detected experimentally. At  $T = 110^\circ\text{C}$ , it is equal to 0.17 mm. During subsequent holding at  $110^\circ\text{C}$ , the bend continues increasing and reaches the maximum magnitude  $y_{\text{max}} = 1.94$  mm in 55 s after the start of hydrogen feeding (shown by letter *d* in Fig. 1a).

After that, the plate begins straightening up, and the deflection decreases. At  $110^\circ\text{C}$ , this second stage is 35 times longer than the first stage (see Fig. 1a). After 2700 s from the onset of the experiment, the plate reaches a stationary state with  $y_{\text{st}} = 0.61$  mm, which remains unchanged during subsequent holding for 1000 s (see Fig. 1a).

At 240 and  $350^\circ\text{C}$  (see Figs. 1b, 1c), the regularities of the time dependence of the form change are qualitatively similar to those described for the temperature of  $110^\circ\text{C}$  (Fig. 1a). However, the quantitative characteristics of the form change were found to be strongly temperature-dependent. The maximum bend of the plate at  $240^\circ\text{C}$  equal to 2.33 mm is 17% higher than  $y_{\text{max}}$  at  $110^\circ\text{C}$ . On the contrary, the maximum bend at  $350^\circ\text{C}$  ( $y_{\text{max}} = 1.75$  mm) is lower substantially; it is 10 and 25% lower than that at 110 and  $240^\circ\text{C}$ , respectively. The time for reaching the maximum bend decreases with increasing temperature; at  $350^\circ\text{C}$ , it is only 5 s.

One other moment is of importance. At  $110^\circ\text{C}$ , the second stage of form change is 35 times longer than the first stage. At 240 and  $350^\circ\text{C}$ , the time of the second stage of saturation is higher than that of the first stage

by a factor of 10 and 20, respectively. This tendency is clearly observed in Figs. 1a–1c.

As the temperature increases, the residual bend of the plate behaves as follows. At  $240^\circ\text{C}$ ,  $y_{\text{st}} = 0.21$  mm, which is approximately one third of that at  $110^\circ\text{C}$  ( $y_{\text{st}} = 0.61$  mm). At  $350^\circ\text{C}$ , the residual bend of the plate is only 0.03 mm, which is only 0.015 of the maximum magnitude at this temperature. Thus, the bend of the plate at  $350^\circ\text{C}$  is found to be almost completely reversible.

Thus, the experiments described above have revealed some tendencies in the effect of temperature on the hydrogen-induced form change of the palladium plate. In accordance with these data, we intended to perform two series of experiments, which are analogous to those described above. The first series of experiments was performed at the same hydrogen pressure, namely, 0.03 MPa. The results are summarized in Table 1; the data obtained in the above-described experiments performed at 110, 240, and  $350^\circ\text{C}$  are also included in it. The second series of experiments (Table 2) was performed at a higher pressure, namely, at 0.09 MPa. An analysis of experimental results given in Tables 1 and 2 confirms the effect of temperature on the form change of the plate at both 0.03 and 0.09 MPa.

Let us now note the most important regularities in the effect of temperature on the hydrogen-induced form change of the palladium plate. As is seen from Tables 1 and 2 (columns 3 and 4), at the first stage of saturation, the time of reaching the maximum bend  $\Delta t_{\text{max}}$  decreases substantially, and the variations of the  $y_{\text{max}}$  magnitude obey an extremal law with increasing

**Table 2.** Effect of temperature on the form change of the palladium plate at  $P_{H_2} = 0.09$  MPa

No	$T, ^\circ\text{C}$	$\Delta t_{\text{onset}}, \text{s}$	$\Delta t_{\text{max}}, \text{s}$	$y_{\text{max}}, \text{mm}$	$\Delta t_{\text{st}}, \text{s}$	$y_{\text{st}}, \text{mm}$	$\frac{y_{\text{st}}}{y_{\text{max}}} \times 100\%$	$n_0, [\text{Pd}/\text{H}]$
	<i>1</i>	<i>2</i>	<i>3</i>	<i>4</i>	<i>5</i>	<i>6</i>	<i>7</i>	<i>8</i>
<i>1</i>	150	6.16	25	2.85	560	0.51	18.0	0.0370
<i>2</i>	170	7.1	15	2.85	1320	0.34	11.8	0.02998
<i>3</i>	200	8.46	10	3.32	1100	0.24	7.1	0.0243
<i>4</i>	220	7.47	13	3.53	170	0.16	4.5	0.0214
<i>5</i>	240	5.12	7	3.48	1080	0.13	3.6	0.0180
<i>6</i>	240	3.74	5.3	3.49	74	0.15	4.4	0.0180
<i>7</i>	260	5.16	6	3.41	300	0.00	0.0	0.0176
<i>8</i>	280	5.8	6	3.06	180	0.03	1.1	0.0162
<i>9</i>	300	5.89	7.3	3.00	600	0.00	0.0	0.0146
<i>10</i>	300	7.96	8	2.83	900	0.09	3.3	0.0146
<i>11</i>	320	7.15	8.4	2.85	300	0.00	0.0	0.0131
<i>12</i>	350	5.01	5	2.75	33	0.07	2.5	0.0109

temperature. At  $P_{H_2} = 0.03$  MPa, the maximum bend  $y_{\text{max}} = 2.4$  mm is reached at  $\sim 200^\circ\text{C}$ , whereas at  $P_{H_2} = 0.09$  MPa, the maximum bend  $y_{\text{max}} = 3.49$  mm occurs at a higher temperature equal to  $\sim 240^\circ\text{C}$ . The time dependence of the straightening of the flexed plate at the second stage of hydrogen saturation also changes substantially with increasing temperature. In this case, the following regularities are revealed from the experimental data summarized in Tables 1 and 2 (columns 5–8).

As the temperature increases to  $350^\circ\text{C}$ , the time of the second stage of hydrogen saturation at  $P_{H_2} = 0.03$  and  $0.09$  MPa ( $\Delta t - \Delta t_{\text{max}}$ ), during which the plate straightens up almost completely, decreases substantially, namely, by factors of 10 and 17, respectively.

In general, the time of complete form change at high temperatures ( $320$ – $350^\circ\text{C}$ ) was found to be quite short. In particular, at  $P_{H_2} = 0.09$  MPa and  $T = 350^\circ\text{C}$ , the plate bends and straightens-up in only  $\sim 30$  s (see Table 2).

At low temperatures, the residual stationary bend ( $y_{\text{st}}$ ) of the plate is 20–30% of the maximum bend. The fact that it remains unchanged for a rather prolonged time is of interest. This is seen clearly from Fig. 1 and is confirmed by the experimental data summarized in Tables 1 and 2.

A very important experimental fact (Tables 1 and 2) consists in the increase in the reversibility of the plate-bend phenomenon with increasing temperature; at  $320$ – $350^\circ\text{C}$ , the phenomenon becomes completely reversible within the accuracy of the method used.

Below, we consider the above experimental data using the modern knowledge on the palladium–hydrogen system.

Note that the introduced hydrogen atoms entail an expansion of the crystal lattice of a metal [2]. This determines the autolocalization of hydrogen atoms near the metal surface during its saturation with hydrogen [3] owing to two causes.

First, attractive forces act between the introduced hydrogen atoms [4]. Therefore, the effective diffusion coefficient of hydrogen in a metal decreases with increasing hydrogen concentration. As was shown in [3], this leads to a deceleration of hydrogen penetration into the metal and to hydrogen localization mainly within a thin near-surface layer [3].

The second cause for the hydrogen localization within the near-surface layer consists in the fact [4] that the hydrogen concentration gradients and corresponding gradients of crystal-lattice dilatation arising during the saturation of metal with hydrogen produce the uphill diffusion of hydrogen, which is directed oppositely to the diffusion flow caused by the concentration gradient (Fick diffusion). It is known that, depending on the experimental conditions, the uphill diffusion can decelerate the hydrogen penetration into a metal [4] and can determine the hydrogen transfer oppositely to its concentration gradient (in particular, during hydride transformations). Under certain conditions [4], a dynamic equilibrium between the Fick and uphill diffusion flows can take place. This phenomenon of thermobaroeelastic diffusive equilibrium has been studied experimentally in [5]. The thermobaroeelastic diffusive equilibrium can prevent the

directional hydrogen transfer between metal regions differing in the hydrogen concentration [5].

Based on the above-described data on the metal–hydrogen systems, conceptions of the metal form change during nonuniform hydrogen saturation of metal have been developed in [1]. The specific character of the hydrogen-induced change of the metal form is determined by the following fundamental peculiarity of the metal–hydrogen systems: during hydrogen saturation, a temporary-gradient (TG) autolocalized “metal–hydrogen” material is formed in the metal, and hydrogen-concentration-induced stresses arise.

Let us consider how this concept “works” in the case of the form-change phenomenon in the palladium plate under study. Remind that the palladium plate of length  $l$  and of thickness  $h$  is subjected to one-sided saturation with hydrogen. During the first stage of saturation with hydrogen, two different layers are formed in the plate. At the entrance surface of the plate, there is formed a growing layer 1 (of thickness and length  $l_1 > l$ ) of the autolocalized TG alloy  $\alpha$ -PdH $_n$ . Layer 2 is the remaining portion of the palladium plate, which is almost unsaturated with hydrogen. Therefore,  $h_2 = h - h_1$ . It is clear that the plate in which interconnected geometrically different layers 1 and 2 are formed becomes bent to maintain the plate integrity and reach stress-compensated state. This is the main mechanical “component” of the bending mechanism of the palladium plate at the first stage of its one-sided saturation with hydrogen.

The physical component of the phenomenon is the matter for further investigations. Now, we only note that, during form change, there are always take place mutually dependent and mutually determined changes in the state of stress of the plate, on the one hand, and in the concentration distribution of the dissolved hydrogen, on the other hand. These synergistically related processes, beyond any question, should substantially affect the kinetics and magnitude of hydrogen-induced form change of metal. In our opinion, this is the essence of the physical component of the form-change mechanism for the palladium plate under study.

The experimental results of this study make it possible to perform an additional phenomenological analysis of the peculiarities of the hydrogen-induced form change of the metal. In particular, the behavior of the temperature dependence of the maximum bending of the plate, which was found experimentally and described above, is of large interest. As was shown earlier [1], it is mainly the following two factors that determine the maximum bending of the plate.

One factor is the hydrogen concentration  $n$  within the autolocalized layer of the temporary-gradient  $\alpha$ -PdH $_n$  alloy. This factor determines the difference in the lengths of layers 1 and 2. In the case of diffusion-controlled process of hydrogen penetration, the hydrogen concentration in layer 1 must be comparable with the equilibrium hydrogen solubility in palladium

( $n \approx n_0$ ) at a given temperature and a given hydrogen pressure. Thus, the equilibrium hydrogen solubility in palladium is an important physical factor determining the maximum bending of the plate and its temperature dependence.

Another important physical factor of the plate bending is the diffusivity of hydrogen in palladium. Indeed, it is just the diffusivity of hydrogen that determines the formation of the autolocalized layer of the temporary-gradient  $\alpha$ -PdH $_n$  alloy and, thus, determines the thicknesses of layers 1 and 2 formed at the moment of the maximum bending of the plate.

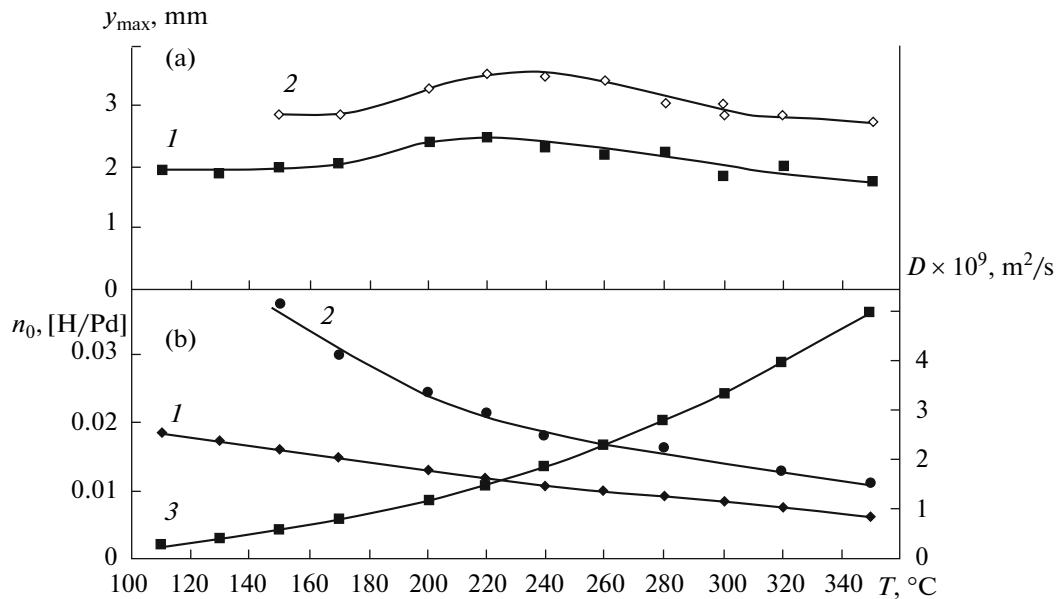
Thus, there are all grounds to assume that it is these two factors, namely, the equilibrium hydrogen solubility in palladium and diffusivity of hydrogen, that mainly determine the maximum bending of the plate. It is, therefore, of interest to compare the temperature dependences of the maximum bending of the plate with those of the coefficient of diffusion and equilibrium hydrogen concentration in palladium at pressures used in our experiment.

Figure 2a summarizes the above experimental data (see Tables 1 and 2) concerning the temperature dependences of the maximum bends ( $y_{\max}$ ) of the palladium plate at  $P_{\text{H}_2} = 0.03$  MPa (curve 1) and  $P_{\text{H}_2} = 0.09$  MPa (curve 2). It is seen clearly that, in both cases, extremal dependences  $y_{\max} = f(T)$  take place; the extremum in curve 2 ( $P_{\text{H}_2} = 0.09$  MPa) is shifted slightly to the high temperature range as compared to that in curve 1 ( $P_{\text{H}_2} = 0.03$  MPa).

Figure 2b summarizes data on the coefficients of hydrogen diffusion in palladium (curve 3) and equilibrium hydrogen solubility in palladium at  $P_{\text{H}_2} = 0.03$  (curve 1) and  $P_{\text{H}_2} = 0.09$  MPa (curve 2), which were calculated by the procedure described in [1].

The comparison of the data given in Figs. 2a and 2b clearly indicates that it is precisely the cooperative effect of two factors,  $D$  and  $n_0$ , that determines the extremal temperature dependence on the maximum bend ( $y_{\max}$ ) of the plate during its one-sided saturation with hydrogen. In this case, it is logical that the highest bends take place at temperatures of 220–260°C, at which the total effect of the physical factors discussed ( $D$  and  $n_0$ ) is obviously maximum.

Let us now consider the experimentally found fact that the hydrogen-induced form change of the palladium plate at high temperatures is completely reversible. It is obvious that the complete reversibility of the phenomenon (at high temperatures) definitely indicates the realization of a coherent mechanism of the plate bending. In the case of such bending, the coherent structure of the arising temporary-gradient  $\alpha$ -PdH $_n$  alloy (layer 1), the coherent structure of layer 2 (stressed palladium), and the coherent structure of the region of matching of layers 1 and 2 are retained. In the case of the realization of the coherent mechanism of form change, the hydrogen concentration (HC)



**Fig. 2.** Temperature dependences of the (a) maximum form change of the plate and (b) coefficient of hydrogen diffusion in palladium (for comments, see text).

stresses induced in the plate do not exceed the proportional elastic limit of palladium and of the gradient  $\alpha$ -PdH<sub>n</sub> alloy. Upon the completion of hydrogen saturation via the coherent mechanism under conditions of reached equilibrium with the gas phase the initial palladium plate becomes transformed into a plate of the equilibrium  $\alpha$ -PdH<sub>n<sub>0</sub></sub> alloy which inherits the annealed state and structure of the initial palladium.

Now, let us consider the experimental fact that at low temperatures relatively high stationary bends ( $y_{\text{st}}$ ) of the plate were found (see Tables 1 and 2). In this case,  $y_{\text{st}}$  decreases with increasing temperature and, at  $T \geq 300^\circ\text{C}$ , the bends become completely reversible.

It was shown in [1] that both during hydrogen saturation and upon mechanical loading up to deflections  $y_{\max} \leq 3\text{--}4$  mm the plate under study bends definitely within the elasticity limits. In other words, at  $y_{\max} \leq 3\text{--}4$ , the maximum stresses induced in near-surface layers of the plate prove to be less than the elastic limit of palladium. In this study, the maximum bends of the plate at all temperatures are  $y_{\max} = 1.75\text{--}1.94$  at  $P_{\text{H}_2} = 0.03$  and  $2.75\text{--}3.53$  mm at  $0.09$  MPa. Thus, we may assume that, under conditions of our experiments, the plate does not undergo plastic deformation. Therefore, the irreversible plastic deformation cannot be considered as the possible cause for the finite stationary bends of the plate (Tables 1 and 2) observed in our experiments.

The other cause for the effect discussed ( $y_{\text{st}}$ ) can be related to the fact that no equilibrium uniform distribution of dissolved hydrogen over the plate thickness is reached during the experiment. Indeed, the mathe-

tical simulation and computer numerical calculations [7] performed in terms of the thermodynamic description of the hydrogen elasticity phenomenon [8] showed that the relative effect of the uphill hydrogen diffusion decreases substantially with decreasing temperature. As a result, the rate of the diffusional approach to equilibrium at low temperatures decelerates substantially. Therefore, we may assume that, at low temperatures, no equilibrium saturation of the plate with hydrogen is reached during our experiment. Correspondingly, a certain gradient of dilatation of the crystal lattice of the  $\alpha$ -PdH<sub>n</sub> alloy and a certain level of hydrogen-concentration stresses are retained in the plate. Accordingly, no complete reversibility of the plate bend is reached during the experiment at low temperatures.

Note one more experimental fact. After reaching the stationary bend  $y_{\text{st}}$ , the plate was usually additionally held in hydrogen from 60 to 2280 s. However, this did not lead to a marked decrease in  $y_{\text{st}}$ . This experimental fact allows us to assert the following hypothesis. At low temperatures, no final leveling-off of the hydrogen concentration to the thermodynamically equilibrium state and no related final straightening-up of the plate are reached at the final stage of saturation of the plate with hydrogen, since a quasi-stationary thermobaroeelastic diffusive equilibrium [4, 5] is set between the nonuniform hydrogen-concentration field and the field produced by residual elastic hydrogen-concentration stresses.

In conclusion, we emphasize that the above hypothesis on the possible realization of the thermobaroeelastic diffusive equilibrium at low tempera-

tures at the final stage of the process of saturation of the plate with hydrogen deserves further systematic theoretical and experimental investigations.

### CONCLUSIONS

(1) Form changes of a one-end-fixed palladium plate  $62 \times 5.5 \times 0.27$  mm in size have been studied during its one-sided saturation with hydrogen at temperatures of 110–350°C. The form change of the plate was confirmed to develop in two stages. During the first stage, the maximum form change (bending) is reached. During the second (more prolonged) stage, the almost complete straightening-up of the plate occurs at high temperatures. At low temperatures, a relatively small residual bend of the plate is retained.

(2) Variations of the maximum bend of the plate with increasing temperature are described by an extremal function; at 220–260°C, the bend reaches a maximum. The maximum bend was shown to be determined by two fundamental properties of the Pd–H system, namely, by the diffusivity of hydrogen and by the equilibrium hydrogen solubility in palladium.

(3) The experimentally found regularities of the hydrogen-induced form change of the palladium plate have been discussed based on the fundamental peculiarities of the metal–hydrogen systems, which consist in the fact that, during metal saturation with hydrogen, there always is formed a temporary-gradient “metal–hydrogen” material and hydrogen-concentration stresses always arise.

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