

Growth Kinetics of $\text{Nd}_2\text{Fe}_{14}\text{B}$ Phase during Hydrogen-Induced Reverse Phase Transformation in $\text{Nd}_2\text{Fe}_{14}\text{B}$ Type Nanocrystalline Magnetic Alloy

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ABSTRACT

The influence of hydrogen pressure on kinetics of growth of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase during hydrogen-induced reverse phase transformations in the industrial $\text{Nd}_2\text{Fe}_{14}\text{B}$ hard magnetic alloy has been studied. It has been determined that, as the temperature and the initial hydrogen pressure increase, a reverse phase transformation significantly accelerates. It has been shown that the kinetics of the reverse phase transformation is controlled by the Fe atoms diffusion and that the rate growth of new $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase increase with increase of initial hydrogen pressure. On the base of the Kolmogorov kinetic theory the kinetic equation describing influence of initial hydrogen pressure on the isothermal kinetic diagram for this transformation has been obtained.

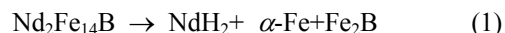
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1. Introduction

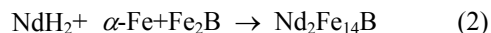
As well known permanent magnet materials of a RE-TM (rare-earth-transition-metal) type are key components of numerous modern devices [1]. There are many processing routes for improving of their magnetic properties [2]. In particular, one from perspective technologies for improvement of magnetic properties of RE-TM type alloys is HDDR-process (Hydrogenation-Decomposition-Desorption-Recombination) [3]. The most significant aspect of the HDDR process is that there is a dramatic change in the microstructure from an initial grain size of typically $\sim 150\text{ }\mu\text{m}$ to a very fine, uniform grain size of about $0.1 - 0.3\text{ }\mu\text{m}$ [3-6]. On magnetisation, the HDDR nanocrystalline powder exhibits an appreciable coercivity and this means that, in its simplest net shape form, the powder can be mixed with a thermosetting resin to produce an isotropic and anisotropic permanent magnet by compression moulding [3-5].

Above-mentioned HDDR-process is based on hydrogen-induced reversible phase transformations in the RE-TM type hard magnetic alloys [6]. The transformation process is based on the formation of a RE-TM (rare-earth-transition-metal) hydride which at higher temperatures has a tendency to disproportionate into a mixture of RE-hydride and iron—direct hydrogen induced

phase transformation that occurs in case of NdFeB-based alloys by the following scheme [6]



Further, at second stage, subsequent desorption of the hydrogen using a vacuum ($\sim 10^{-2}$ Torr) at high temperatures from decomposed alloy leads to recombination into initial phase of $\text{Nd}_2\text{Fe}_{14}\text{B}$ —reverse hydrogen induced phase transformation by the following scheme in NdFeB-based alloys [6]



The carrying out of these type phase transformations lead to formation of a fine-grained ferromagnetic phase $\text{Nd}_2\text{Fe}_{14}\text{B}$ that allows to obtain permanent magnets with a high coercive [4,5].

Earlier the effect of the hydrogen pressure on the kinetics of hydrogen-induced direct phase transformations in RE-TM type alloys has been studied systematically in [7-10] and was established that phase transformation process accelerates as hydrogen pressure increase.

Thus, the main aim of this paper is to establish the influence of hydrogen pressure on kinetics of subsequent hydrogen-induced reverse phase transformations proceed by Equation (2) in the $\text{Nd}_2\text{Fe}_{14}\text{B}$ type alloys and to describe kinetics features within a framework of classical

kinetic theory of phase transformations in condensed state.

2. Experimental

Studies of the reverse phase transformations have been carried out on special hydrogen-vacuum equipment using a Sadikov's type magnetometer [9,10]. The industrial $R_2\text{Fe}_{14}\text{B}$ type alloy ($R_{36.4}\text{Fe}_{62.45}\text{B}_{1.15}$, where R is mixture of rare-earth metals—33% Nd, 2% Pr, 0.9% Ce and 0.5% Dy, weight %) samples in the form of powder with average grain size $\sim 100\text{ }\mu\text{m}$ ($\sim 1.25\text{ g}$) were placed into the reaction chamber, which was evacuated to a pressure of $\sim 1\text{ Pa}$. Then, the samples were heated under vacuum to the desired temperature. After establishing isothermal conditions, the reaction chamber was filled with hydrogen under selected pressures ranging from 0.1 to 0.2 MPa. The direct transformation was continuously monitored under isothermal conditions by measuring the amount of newly formed ferromagnetic phases ($\alpha\text{-Fe}$ and Fe_2B) according to scheme (1). After completion of the direct

transformation, from moment of hydrogen evacuation from reaction chamber it lead to the reverse phase transformation evolution, *i.e.* recombination of decomposed phases into $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase according to scheme (2). The development of the reverse transformations was then followed by continuously monitoring the decreasing amount of the ferromagnetic phases in the sample. The resulting data were plotted as isothermal kinetic diagrams of the reverse transformations occurring in the $R_2\text{Fe}_{14}\text{B}$ compound.

3. Results and Discussion

The isothermal kinetic diagrams of the reverse phase transformation in the industrial $R_2\text{Fe}_{14}\text{B}$ alloy at various temperatures and initial hydrogen pressures are shown in **Figure 1**. As follows from **Figure 1** if the reverse phase transformation starts from higher initial hydrogen pressure it leads to increase of phase transformation evolution for all transformation temperatures. In general, a

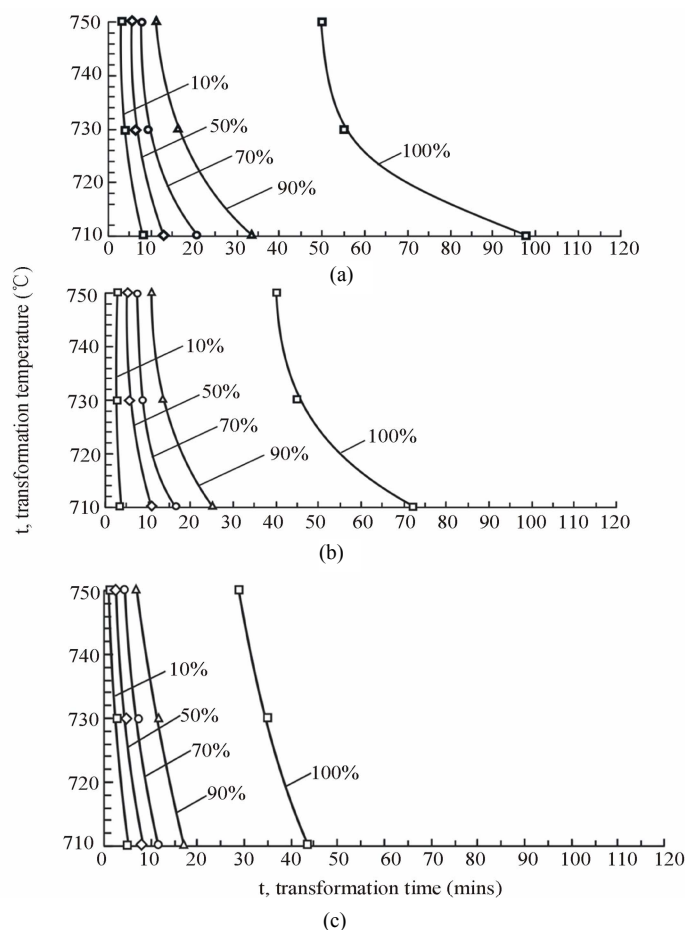


Figure 1. The isothermal kinetic diagrams for hydrogen induced reverse phase transformation in $R_2\text{Fe}_{14}\text{B}$ alloy, where T is the isothermal exposure temperature, t is the transformation time and 10, 30, 50, 70, 90 100% is the degrees of the reverse transformation at different initial hydrogen pressure: (a) – 0.10 MPa; (b) – 0.15 MPa; (c) – 0.20 MPa.

twofold increase of the initial hydrogen pressure (from 0.1 MPa up to 0.2 MPa) results in an acceleration of the reverse phase transformation evolution in the R₂Fe₁₄B alloy in ~1.5–2 times.

As can be seen from Equation (2) for transformation of this type needs diffusion of alloy's components and in fact earlier on a base of kinetic, TEM and X-ray diffraction studies during reverse phase transformation in Nd₂Fe₁₄B type alloys has been showed that transformations of this type maybe classified as diffusive phase transformation in solid state and that the reverse transformation process proceeds by the nucleation and growth mechanism [11,12]. Because of this, according to detailed TEM investigations [12] at first NdH₂ phase dissociates into Nd due to desorption of H₂ and then α -Fe diffuses into Nd. The Fe₂B grains act as boron carriers and due to their small size and random distribution there are short transfer-lengths for the boron atoms during recombination. Finally the disproportionated mixture recombines to the thermodynamically more stable Nd₂Fe₁₄B phase.

Therefore, it is obviously that from viewpoint of classical kinetic theory of phase transformations in condensed state above-described hydrogen-induced reverse phase transformation in Nd₂Fe₁₄B type alloys proceeds by the nucleation and growth mechanism. Further, it is possible to find an effective activation energy of phase transformation process in accordance with Becker-Döring model of nucleation kinetics [13,14] if plots dependence $\ln t$ on $1/T$ where t is the time, which is needed for reaching of some degree of transformation and T is the transformation temperature. With this goal experimental data from **Figure 1** were re-plotted in co-ordinates $\ln t$ versus $1/T$ which are shown in **Figure 2**. The obtained values of effective energy of phase transformation for various hydrogen pressures and degrees of transformation are given in **Tables 1**.

As can be seen the from **Table 1** all obtained values of effective activation energy have good agreement by order of magnitude with activation energy for iron atoms diffusion in α -phase of Fe ($Q_{\text{Fe}} = 259.54 \div 284.2$ kJ/mol [15,16]). Therefore, it is really possible to consider that evolution process of reverse phase transformation is con-

trolled by iron atoms diffusion to new centres of Nd₂Fe₁₄B phase. On the other hand, it is obviously that the driving force of this transformation is dissociation of NdH₂ phase due to desorption of H₂. Thus, in accordance with above-described model transformation let's obtain then kinetic equation for hydrogen induced reverse phase transformation in Nd₂Fe₁₄B type alloy taking into account influence of hydrogen pressure at direct phase transformation stage on reverse transformation kinetics.

In accordance with well known Kolmogorov kinetic theory of phase transformation in condensed state [17] the volume of transformed area during reverse phase transformation can be written as follows:

$$\xi = \frac{V(t)}{V_0} = 1 - \exp\left(-\frac{\pi}{3} I \omega^3 t^4\right), \quad (2)$$

where $V(t)$ is the transformed area volume at time moment t , V_0 is the initial untransformed volume, I is the nucleation rate of centres of new Nd₂Fe₁₄B phases, ω is the rate of growth of a new Nd₂Fe₁₄B phase.

On the other hand, rate of nucleation I of new Nd₂Fe₁₄B phases centers in condensed systems in accordance with Turnbull-Fisher model [18,19] in case of diffusion-controlled growth is

$$I = \gamma \frac{RT}{h} e^{-\frac{\Delta G + Q}{RT}}, \quad (3)$$

where G is the energy necessary for formation of Nd₂Fe₁₄B critical nucleus, Q is the activation energy diffusion of Fe atoms to centres of new Nd₂Fe₁₄B phases, $\gamma = 10^{-4}$ mol/m³ [20], R is the gas constant, h is the Planck constant, T is transformation temperature.

Then, substitute equation (3) into (2) we can obtain kinetic equation for volume of the transformed area ξ in dependence on t transformation time and transformation temperature T :

$$\xi(t) = 1 - \exp\left(-\frac{\pi}{3} \gamma \frac{RT}{h} e^{-\frac{\Delta G + Q}{RT}} \omega^3 t^4\right) \quad (4)$$

As a rule, phase transformation kinetics for practical application describes by curves showing time transformation t needs for reaching some degree of transformation ξ in dependence on transformation temperature T

Table 1. The effective activation energy of hydrogen induced reverse phase transformation in R₂Fe₁₄B alloy for various initial hydrogen pressures P and degrees of phase transformation ξ .

ξ , degree of transformation	$P = 0.1$ MPa, initial hydrogen pressure	$P = 0.15$ MPa, initial hydrogen pressure	$P = 0.2$ MPa, initial hydrogen pressure
0.5	198.31 ± 7.51 kJ/mol	181.24 ± 6.78 kJ/mol	214.23 ± 8.05 kJ/mol
0.7	225.29 ± 7.95 kJ/mol	177.83 ± 7.96 kJ/mol	216.62 ± 8.23 kJ/mol
0.9	224.46 ± 8.14 kJ/mol	224.99 ± 7.96 kJ/mol	198.96 ± 8.21 kJ/mol

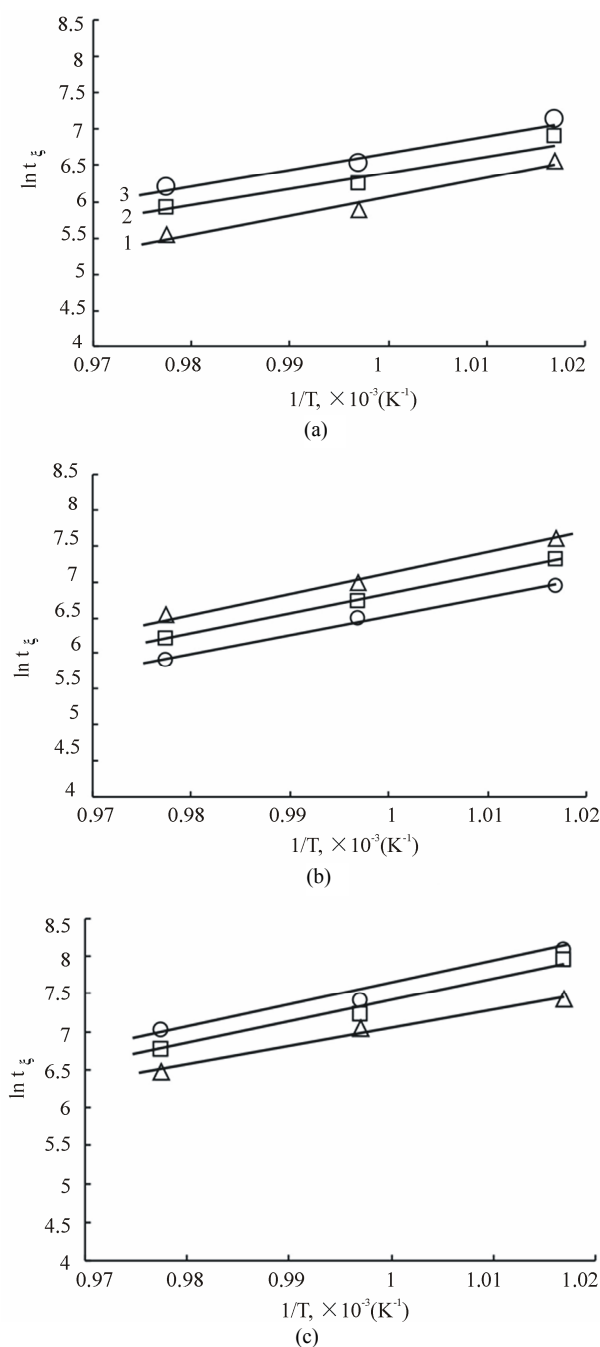


Figure 2. The dependence of $\ln t_\xi$ versus $1/T$ for hydrogen induced reverse phase transformation in $R_2\text{Fe}_{14}\text{B}$ alloy for degrees of transformation –50% (a), 70% (b) and 90% (c) and following initial hydrogen pressure: 1 - 0.20 MPa; 2 - 0.15 MPa; 3 - 0.10 MPa.

[22]. Thus, in this case Equation (4) has been written in following form:

$$t(\xi, T) = \left(\frac{3h}{\pi\gamma RT} \right)^{\frac{1}{4}} \frac{(-\ln(1-\xi))^{\frac{1}{4}}}{\omega^{\frac{3}{4}}} e^{\frac{U}{4RT}}. \quad (5)$$

Further, in isothermal conditions the rate of nucleation I describing by Equation (3) is constant in accordance to classical theory of nucleation rate in condensed systems because W and Q as a rule are constants for same alloy [20,22]. Therefore, we can believe that the rate of nucleation of Nd₂Fe₁₄B phase is not depends on initial

hydrogen pressure. Let's determine further rate of nucleation I and rate of growth of a new Nd₂Fe₁₄B phase ω believed that the activation energy at transition of atoms through interface of phases Q is equal in our case to activation energy of diffusion of Fe atoms in α -Fe phase $Q = 259.54$ kJ/mol [16] and energy necessary for formation of Nd₂Fe₁₄B critical nucleus $\Delta G = 314.1$ kJ/mol in accordance with data from [23]. Thus, using Equation (3) and kinetic data from **Figure 1** were calculated values of rate of nucleation I and rate of growth of Nd₂Fe₁₄B phase ω during phase transformation for various hydrogen pressures and temperatures for degree of transformation $\xi = 0.9$ which are given in **Tables 2**.

In **Figure 3** is presented dependence growth rate of Nd₂Fe₁₄B phase versus hydrogen pressures P plotted on the base of data from **Table 2**. As follows from **Figure 3**, the rate of growth ω increase with increase of hydrogen pressure at all temperatures.

Further, in accordance with Lyubov and Hillert kinetic approach [20,21] for diffusion-controlled growth we assume that dependence rate of growth ω on hydrogen pressure P and transformation temperature T can be approximated by following type function:

$$\omega = M \frac{\Delta F - \Delta F_s}{RT} e^{-\frac{U(P)}{RT}},$$

where M is proportional to the mobility coefficient of the interface of growing Nd₂Fe₁₄B phase, ΔF is the molar difference of the free energies of initials and final phases, ΔF_s is the driving force of phase transformation which in our case is proportional to the dissociation energy of NdH₂ phase, $U(P)$ is the activation energy transition of Fe atoms across the interface of Nd₂Fe₁₄B phase depending on hydrogen pressure.

Then, let's denote

$$M \frac{\Delta F - \Delta F_s}{RT} = A(P)$$

Further, activation energy transition of Fe atoms across interface of phases $U(P)$ in dependence on initial hydrogen pressure P has been approximated using data from **Table 2** by following function:

$$U(P) = (113.08 \times 10^3 - 78.77 \times 10^3 \times P)$$

Finally, the rate of growth ω depends on initial hydrogen pressure P in R₂Fe₁₄B alloy by following type equation:

$$\omega = A(P) e^{-\frac{113.08 \times 10^3 - 78.77 \times 10^3 \times P}{RT}}, \quad (6)$$

where $A(P)$ is the preexponential function that depends on hydrogen pressures P , which are presented in **Table 3** for various hydrogen pressure.

As can be seen from **Table 3**, $A(P)$ function increase with increase of initial hydrogen pressure that is possible if the mobility coefficient of interface M will increase. In our case hydrogen atoms can be considered as interstitial atoms and increase of their concentration lead to increase of diffusion of matrix atoms (Fe, Nd, B atoms in our case) as was shown in [24-26]. Moreover, the hydrogen atoms concentration was larger in case if reverse transformation was started when hydrogen pressure was larger too and in this case it lead to increase of diffusion of matrix atoms and it correspondingly lead to increase of mobility of interface M . On the other hand, $U(P)$ decrease with hydrogen pressure increase. Hence, the rate growth of new Nd₂Fe₁₄B phase ω depends on two main factors: increasing of mobility coefficient of interface M with hydrogen pressure increase and decreasing of activation energy transition of Fe atoms through interface of Nd₂Fe₁₄B phase $U(P)$ with hydrogen pressure increase. Finally, substitute Equation (6) into Equation (5) we can written the final equation describing dependence transformation time t needs for reaching some degree of transformation ξ on hydrogen pressures P and transformation temperature T :

$$t(\xi, T, P) = \left(\frac{3h}{\pi \gamma RT} \right)^{\frac{1}{4}} \frac{(-\ln(1-\xi))^{\frac{1}{4}}}{\left(A(P) e^{-\frac{113.08 \times 10^3 - 78.77 \times 10^3 \times P}{RT}} \right)^{\frac{3}{4}}} e^{\frac{\Delta G + Q}{4RT}}, \quad (7)$$

Table 2. The rate of nucleation I and rate of growth of a new Nd₂Fe₁₄B phase ω of phase transformation determined for various hydrogen pressures P and transformation temperatures T for degree of transformation $\xi = 0.9$ in R₂Fe₁₄B alloy.

T, temperature (K)	I, nucleation rate (m ⁻³ ·s ⁻¹)	ω , rate of growth at $P = 0.1$ MPa (m·s ⁻¹)	ω , rate of growth at $P = 0.15$ MPa (m·s ⁻¹)	ω , rate of growth at $P = 0.2$ MPa (m·s ⁻¹)
983	0.392×10 ³	3.448×10 ⁻⁶	5.125×10 ⁻⁶	6.064×10 ⁻⁶
1003	1.623×10 ³	4.336×10 ⁻⁶	5.939×10 ⁻⁶	7.114×10 ⁻⁶
1023	6.357×10 ³	8.895×10 ⁻⁶	10.291×10 ⁻⁶	12.42×10 ⁻⁶

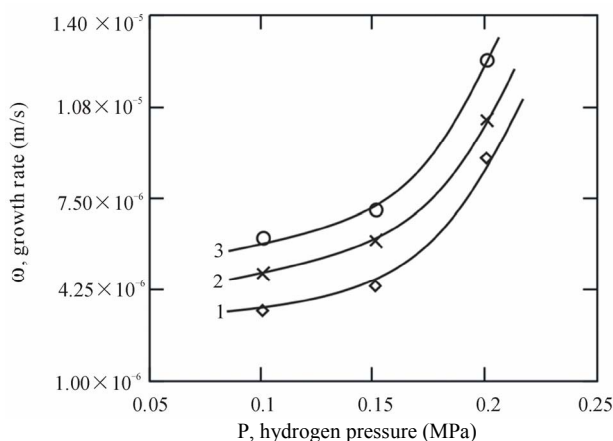


Figure 3. The growth rate of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase ω versus hydrogen pressure P for various transformation temperature: 1 - 710°C; 2 - 730°C; 3 - 750°C for degree of transformation $\xi = 0.9$ in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy.

Table 3. The preexponential function $A(P)$ at different initial hydrogen pressures P determined for degree of reverse hydrogen induced transformation $\xi = 0.9$ in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy.

P , initial hydrogen pressure (MPa)	$A(P)$, the preexponential function in Equation (6)
0.10	94.73×10^{-2}
0.15	97.41×10^{-2}
0.20	98.76×10^{-2}

In **Figure 4** is shown the isothermal kinetic diagram for hydrogen induced reverse phase transformation in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy calculated on the base of Equation (7) and data from **Table 3**.

Thus, it is possible to conclude that obtained Equation (7) well describes experimental results by influence of value of initial hydrogen pressure on kinetic of reverse phase transformation in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy in terms of two main kinetic factors, *i.e.* increasing of mobility coefficient of interface M with hydrogen pressure increase and decreasing of activation energy transition of Fe atoms across interface of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase $U(P)$ with hydrogen pressure increase.

4. Conclusions

The effect of initial hydrogen pressure on the kinetics of growth of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase during hydrogen induced reverse phase transformations in the industrial $\text{R}_2\text{Fe}_{14}\text{B}$ alloy has been studied.

It has been established that, as the temperature and the initial hydrogen pressure increase it lead to considerable acceleration of evolution of reverse phase transformation. It has been obtained that kinetics of reverse phase transformation process is controlled by Fe atoms diffusion

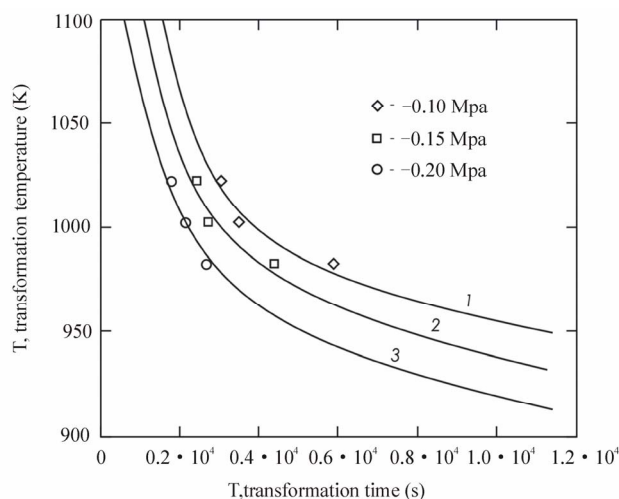


Figure 4. The isothermal kinetic diagram for hydrogen induced reverse phase transformation in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy calculated by Eq. (7) for degree of transformation $\xi = 0.999$ for various hydrogen pressure: 1 – 0.10 MPa; 2 – 0.15 MPa; 3 – 0.20 MPa. Points are experimental data from Figure 1 for following hydrogen pressures.

and the rate growth of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase ω increase with increase of initial hydrogen pressure.

On the base of Kolmogorov kinetic theory of evolution of phase transformation in condensed state the equation describing the isothermal kinetic diagrams of such type of transformations has been obtained. It has been shown that effect of initial hydrogen pressure on kinetics of hydrogen induced reverse transformations in $\text{R}_2\text{Fe}_{14}\text{B}$ alloy the may be described by following type equation:

$$t(\xi, T, P) = \left(\frac{3h}{\pi\gamma RT} \right)^{\frac{1}{4}} \frac{(-\ln(1-\xi))^{\frac{1}{4}} e^{\frac{\Delta G + Q}{4RT}}}{\left(A(P) e^{\frac{113.08 \times 10^3 - 78.77 \times 10^3 \times P}{RT}} \right)^{\frac{3}{4}}}$$

where $t(\xi, T, P)$ is the transformation time for various degree of transformation ξ at different temperature T and initial hydrogen pressure P , ΔG is the energy necessary for formation of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase critical nucleus, Q is the activation energy diffusion of Fe atoms to centers of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase, $\gamma = 10^{-4}$ mol/m³, R is the gas constant, h is the Planck constant, T is transformation temperature, $A(P)$ is the preexponential function depending on hydrogen pressure, P is the initial hydrogen pressure.

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