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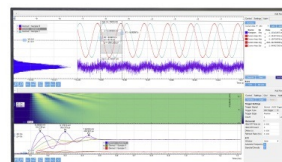


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Kinetics of Hydrogen-induced Phase Transformations in Nd₂Fe₁₄B Type Hard Magnetic Alloy for Permanent Magnets

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Abstract. The effect of hydrogen pressure on kinetics of hydrogen-induced direct phase transformation in the industrial Nd₂Fe₁₄B type ($R_{36.4}Fe_{62.45}B_{1.15}$, where R – Nd, Pr, Ce, Dy) hard magnetic alloy for powder permanent magnets has been studied. It has been determined that, as the hydrogen pressure increase, a direct phase transformation significantly accelerates. It has been shown that the kinetics of direct phase transformation controlled by the Fe atoms diffusion and that the rate growth of new Nd₂Fe₁₄B phase increase with increase of initial hydrogen pressure. On the base of the Kolmogorov's and Lyubov's kinetic models the kinetic equation for describing of hydrogen pressure influence on the kinetic of this transformation in Nd₂Fe₁₄B type alloys has been presented.

INTRODUCTION

It is known that permanent magnet materials are key components of electronic, data processing, medical devices and others important modern devices. In particular, permanent magnets with the best magnetic properties are produced from R₂Fe₁₄B compounds (R – Nd, Pr, Ce, Dy etc.) type alloys [1,2]. New technology known as the HDDR-process (Hydrogenation–Disproportionation–Desorption–Recombination) is based on hydrogen-induced direct and reverse diffusive phase transformations in the R₂Fe₁₄B type hard magnetic alloys is one of the perspective technologies for improvement magnetic properties of R₂Fe₁₄B type powder permanent magnets [1,3-5].

In particular, interaction of R₂Fe₁₄B type alloy with hydrogen leads to direct transformation when initial alloy decomposes into α -Fe, Fe₂B and RH₂ [3-5]. Then, evacuation of hydrogen leads to evolution of a reverse transformation with recombination of an initial R₂Fe₁₄B alloy but with a significantly modified structure and with improved of magnetic characteristics of powder permanent magnets [3-5]. Earlier the effect of the hydrogen pressure on the kinetics of hydrogen-induced direct phase transformations in industrial R₂Fe₁₄B alloy ($R_{36.4}Fe_{62.45}B_{1.15}$, where R is mixture of rare-earth metals – 33% Nd, 2% Pr, 0.9% Ce and 0.5% Dy, weight %) has been studied systematically [6,7]. Therefore, the main goal is to describe the influence of hydrogen pressure on kinetics of hydrogen-induced direct diffusive phase transformations in the R₂Fe₁₄B type alloys within a framework of kinetic theory of phase transformations in solid state.

MATERIALS AND METHODS

In experiments the industrial $R_2Fe_{14}B$ type alloy ($R_{36.4}Fe_{62.45}B_{1.15}$, 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 $\sim 100 \mu m$ (~ 1.25 g) were placed into the reaction chamber, which was evacuated to a pressure of ~ 1 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. Studies of the direct phase transformations have been carried out on special hydrogen-vacuum equipment using a Sadikov's type magnetometer [8,9]. The direct transformation was continuously monitored under isothermal conditions by measuring the amount of newly formed ferromagnetic phases (α -Fe and Fe_2B). The effect of the hydrogen pressure on the kinetics of hydrogen-induced direct phase transformations in industrial $R_2Fe_{14}B$ alloy (R – 33% Nd, 2% Pr, 0.9% Ce and 0.5% Dy, weight %) has been studied experimentally in works [6,7].

RESULTS AND DISCUSSION

The kinetic curves of direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy obtained earlier [6,7] at $710^\circ C$ and hydrogen pressure of 0.10, 0.15 and 0.20 MPa are shown in Figure 1. As seen the phase transformation is completed in 1.59×10^4 s at hydrogen pressure of 0.10 MPa and 1.2×10^4 s at hydrogen pressure of 0.15 MPa. An increase in hydrogen pressure to 0.20 MPa results in completion of the direct transformation in 0.78×10^4 s. Thus, as follows from Fig. 1, a twofold increase of hydrogen pressure lead to significant acceleration of phase transformation developing in two times approximately. Thus, interaction of $R_2Fe_{14}B$ type hard magnetic alloy with hydrogen leads to direct transformation evolution when initial alloy decomposes into rare-earth hydride RH_2 phase, α -Fe phase and Fe_2B phase by the following phase scheme [3-5]:



It should be noted that earlier on the basis of investigation of microstructure features in $Nd_2Fe_{14}B$ type alloys [2,3,10-13] it is established that the main phases in decomposed alloy is earth hydride NdH_2 phase and α -Fe phase with small content of iron boride Fe_2B phase.

Therefore, we can believe in our case that evolution process of transformation is controlled by two main processes namely nucleation and growth process of the two main phases: RH_2 rare-earth hydride phase and α -Fe phase. Further, in accordance with above described model let's obtain kinetic equations for direct hydrogen induced phase transformation in $R_2Fe_{14}B$ type alloys. According to the Kolmogorov's kinetic theory of phase transformation in solid state [14,15], the volume of the transformed area f in dependence on t transformation time can be written as

$$f(t) = 1 - \exp \left[- \int_0^t I(t) \varphi(t - \tau) dt \right], \quad (2)$$

where $I(t)$ is the nucleation rate of centres of new phases at time t , $\varphi(t)$ is the volume of this nucleation centre at time t , τ is the nucleation moment of centre of a new phase. In our case of isothermal conditions it is believed that $I(t) = const$.

Then, in general case, according to the Lyubov's kinetic theory approach [16] integral in Equation (2) we can divide in two integrals: from 0 to τ and from τ to t , where τ is time which is necessary for growth of nucleus of new phase up to critical size. Therefore, Eq. (2) maybe written in following form:

$$f(t) = 1 - \exp \left[- \left(\int_0^\tau I \varphi_1(t - \tau) dt + \int_\tau^t I \varphi_2(t - \tau) dt \right) \right], \quad (3)$$

where τ is time which is necessary for growth of nucleus of new phase up to critical size, $\varphi_1(t)$ and $\varphi_2(t)$ is the volume of new phases of RH₂ and α -Fe nucleation centres at time moment t , correspondingly. Further, growth of volume of a spherical centre of RH₂ phase $\varphi_1(t)$ may be written as:

$$\varphi_1(t) = \frac{4}{3}\pi r_1^3(t), \quad (4)$$

where $r_1(t)$ is the radius of this RH₂ phase centre at time t .

Further, nucleation process of RH₂ phase we can be considered as in single-component system taking into account of high diffusive mobility of hydrogen atoms to rare-earth atoms in R₂Fe₁₇ alloy and therefore by theory of absolute reaction rates [16,17] radius $r_1(t)$ of RH₂ centre at time t :

$$r_1(t) = \frac{\pi d^4 \Delta g}{9h} e^{-\frac{U}{RT} t}, \quad (5)$$

where Δg is change of free energy at formation of volume unit of new phase, U is activation energy of atoms at transition through interface of phases, d is diameter of diffusing atom (hydrogen atoms in our case), h is the Planck constant, R is the gas constant, T is the transformation temperature, t is some time moment.

Then, by analogy with above, growth of volume of a spherical centre of α -Fe phase $\varphi_2(t)$ may be written as:

$$\varphi_2(t) = \frac{4}{3}\pi r_2^3(t), \quad (6)$$

where $r_2(t)$ is the radius of α -Fe phase centre at time t .

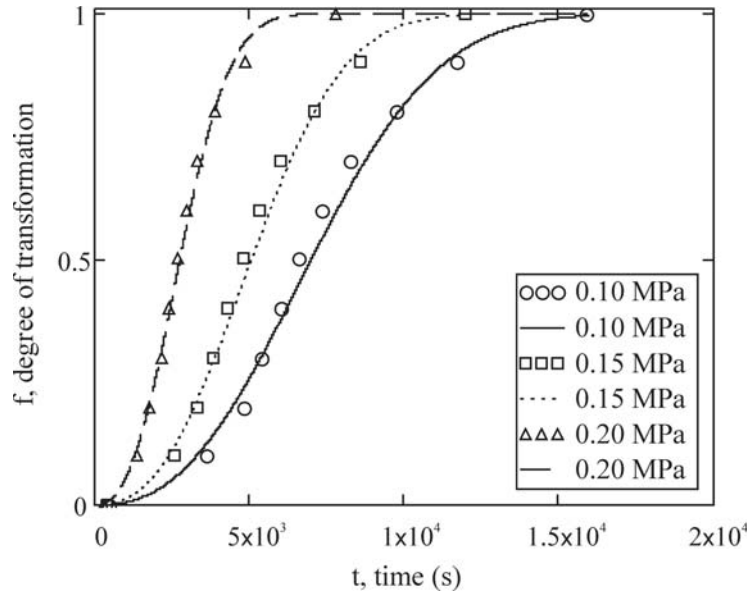


FIGURE 1. Kinetic curves of direct hydrogen-induced phase transformation in R₂Fe₁₄B hard magnetic alloy at 710°C and hydrogen pressure of 0.10, 0.15 and 0.20 MPa (points – experimental data, curves – theoretical calculations).

In view of diffusive-controlled character of these type transformations that was established earlier [5] and therefore in case of diffusive-controlled transformations boundary of new phase moves by parabolic law [15,16,18] and $r_2(t)$ may be written in following form:

$$r_2(t) = 2\beta\sqrt{Dt}, \quad (7)$$

where $D = D_0 e^{-\frac{Q}{RT}}$ is the diffusion coefficient of Fe atoms, Q is the activation energy of Fe atoms diffusion, R is the gas constant, β is kinetic parameter, T is the transformation temperature, t is some time moment. Further, rate of nucleation I of new phase's centers in condensed systems in accordance with Turnbull-Fisher model [16,19] is

$$I = \gamma \frac{RT}{h} e^{-\frac{W}{RT}} e^{-\frac{U}{RT}}, \quad (8)$$

where W is the energy necessary for formation of critical nucleus, U is the activation energy at transition of atoms through interface of phases, $\gamma=10^{-4}$ mol/m³ [16], R is the gas constant.

Then, substitute Eqs. (4)-(8) in (2) we can obtain kinetic equation for volume of the transformed area f in dependence on transformation time t and temperature T :

$$f(t) = 1 - \exp \left[- \frac{\pi^4 d^{12} \Delta g^3 RT \gamma}{3^7 h^4} e^{-\frac{W+4U}{RT}} t_c^4 - \frac{64\pi RT \gamma}{15h} \beta^3 D_0^{\frac{3}{2}} e^{-\frac{W+U+\frac{3}{2}Q}{RT}} (t^{5/2} - \tau^{5/2}) \right], \quad (9)$$

where t_c is time which is equal to time for nucleus growth of new phase up to critical size.

In general case Eq. (9) describe kinetics of direct hydrogen-induced transformations in Nd₂Fe₁₄B type alloys which has C-shaped type of T-T-T diagram [5] when transformation proceeds with high rate in narrow interval of temperatures ~600-700°C where there is an optimum combination of the growth rates of a-Fe phase and NdH₂ phase. If then consider the possible extreme case when transformation time t is much more than t_c and $\tau (t \geq t_c \geq \tau)$ and the direct hydrogen-induced phase transformation in R₂Fe₁₄B type alloys is developing in low temperature region (600-750°C) [5] where transformation process is determined by the diffusion-controlled growth of new phases [20] we can obtain the following equation:

$$f(t) = 1 - \exp \left[- \frac{64\pi RT \gamma}{15h} \beta^3 D_0^{\frac{3}{2}} e^{-\frac{W+U+\frac{3}{2}Q}{RT}} t^{5/2} \right] \quad (10)$$

It should be noted that exponent at time in Eq. (10) $n=5/2$ and it corresponds diffusion-controlled growth of new phases [15,16] whereas in the Kolmojgorov's theory [14] exponent at time $n=3+4$. It is known that if the experimental results reconstruct in coordinates $\ln[-\ln(1-f)]$ versus $\ln t$, it is possible to establish a kinetic constant n that gives us some important information about the most probable mechanisms of transformations. With this aim the experimental data from Fig. 1 were re-plotted in coordinates $\ln[-\ln(1-f)]$ versus $\ln t$ as it shown in Fig. 2. At hydrogen pressure of 0.10 MPa the exponent at time $n=3.1$, at hydrogen pressure of 0.15 MPa the exponent at time $n=3.0$ and $n=2.7$ at hydrogen pressure of 0.20 MPa. Thus, average experimental value of exponent at time $\langle n \rangle = 2.9$ that is close to $n=5/2$ in Eq. (10). All calculated values of exponent at time n at various hydrogen pressures for direct hydrogen-induced phase transformation in R₂Fe₁₄B hard magnetic alloy are shown in Table 1. Further, for the following analysis it is necessary to determine parameters in equation (10). In our case we can believed that Q is the activation energy of Fe atoms diffusion (in our case we can take on following data for diffusion of Fe atoms in Rare-Earth metals – $Q=250$ kJ/mol, $D_0=1$ mm²/s [21-23]), U is the activation energy of hydrogen atoms at transition of atoms through interface of RH₂ phases which as it was shown earlier [5,20] is equal to energy of RH₂ hydride phase formation nucleus ($\Delta H=-180$ kJ/mol for NdH₂ phase [25]). The kinetic parameter in equation (10) $\beta=10^{-3}$ in accordance with kinetic data for Nd₂Fe₁₄B alloy [20]. In general case we can believed that at constant temperature (710°C) and in a narrow hydrogen pressure (0.1-0.2 MPa) range the activation energy of Fe atoms diffusion and activation energy of hydrogen atoms at transition of atoms through interface of RH₂ phases are constant.

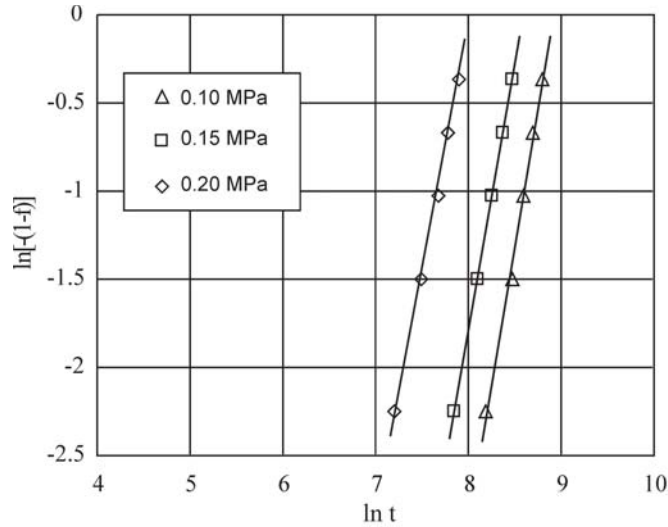


FIGURE 2. $\ln[-\ln(1-f)]$ dependence on $\ln t$ for direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy.

Then, using above-mentioned parameters and experimental data were calculated the free energy of critical nucleus of α -Fe phase formation W for various hydrogen pressure which are shown in Table 1. As follows from Table 1 value of energy of critical nucleus of α -Fe phase formation decrease from 293.9 kJ/mol to 274.9 kJ/mol with increase of hydrogen pressure during phase transformation process from 0.1 to 0.2 MPa. Because of this the energy of critical nucleus of α -Fe phase formation W in $R_2Fe_{14}B$ is similar with same energies in Sm_2Fe_{17} (239.9 kJ/mol) and Y_2Fe_{17} (353.4 kJ/mol) [25].

It should be noted that the above-mentioned values of the energy have reasonable order of magnitude for energy of critical nucleus phase formation in solid body [15]. In turn, the decrease of energy of critical nucleus of α -Fe phase formation with increase of hydrogen pressure leads to an increase in the rate of the nucleation I in accordance with Eq. (7) and therefore the transformation accelerates. As hydrogen pressure increase it leads to increase of hydrogen concentration [26] and consequently to increasing of probability formation of RH_2 hydride phase in decomposing $R_2Fe_{14}B$ alloy and the released Fe atoms diffuse to the growing centres of α -Fe phase and developing of phase transformation accelerates.

Then on the base of Eq. (10) and calculated data from Table 1 the kinetic curves of direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy at 710°C and hydrogen pressure of 0.10, 0.15 and 0.20 MPa have been plotted.

TABLE 1. Exponent at time n and estimated energy values of critical nucleus of α -Fe phase formation in Eq. (10) at various hydrogen pressure for direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy.

Hydrogen pressure (MPa)	Exponent at time in Eq. (9) n (calculated from $\ln[-\ln(1-f)]$ dependence on $\ln t$ in Fig. 2)	Estimated energy of critical nucleus of α -Fe phase formation W (kJ/mol) in Eq. (10)
0.10	3.0	293.9
0.15	3.1	287.3
0.20	2.7	274.9

Fig. 1 shows calculated kinetic curves of direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy. As can be seen from this Fig. 1, the calculated curves well approximate experimental kinetic data of hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy at 710°C and various hydrogen pressures of 0.10, 0.15 and 0.20 MPa. Then, from a practical point of view for HDDR-treatment of $R_2Fe_{14}B$ hard magnetic alloys for powder permanent magnets production need to know hydrogen treatment time. For realization of this goal data

from Table 1 for estimated energy of critical nucleus of α -Fe phase formation W were plotted in dependence of hydrogen pressure P (see Fig. 3). As can be seen from Fig. 3 energy of critical nucleus of α -Fe phase formation is linearly dependent on hydrogen pressure. With taking into account dependence of energy of critical nucleus of α -Fe phase formation W from hydrogen pressure P during direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ alloy can be approximated by following equation:

$$W = -1.84 \cdot 10^5 \cdot P + 3.128 \cdot 10^5, \quad (11)$$

where P is the hydrogen pressure (MPa).

Further, using Eq. (11) energies of critical nucleus of α -Fe phase formation W for high hydrogen pressures range from 0.3 up to 0.7 MPa were calculated (see Table 2).

Then on the base of Eq. (10) using data from Table 2 were calculated the kinetic curves of direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy (constant temperature - 710°C) for high hydrogen pressure range of 0.3-0.7 MPa (see Fig. 4). As can be seen from Fig. 4, the calculated curves predict a significant increase of transformation's rate as hydrogen pressure rise.

TABLE 2. The energy values of critical nucleus of α -Fe phase formation W for various hydrogen pressure P for direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy.

P , hydrogen pressure during phase transformation (MPa)	The energy of critical nucleus of α -Fe phase formation W (kJ/mol) calculated by Eq. (11)
0.3	257.60
0.5	220.86
0.7	184.00

The similar effect also was established earlier in $Nd_{15}Fe_{77}B_8$ hard magnetic alloy in hydrogen pressure range of 0.1-0.7 MPa [27,28] and in (Tb,Dy)-Fe type alloys ($TbFe_2$, Tb_2DyFe_6 , $TbDyFe_4$, $DyFe_2$) in hydrogen pressure interval of 1.3-16.2 kPa [29].

In addition, in $Nd_{16}Fe_{76}B_8$ hard magnetic alloy has been established that kinetics of decomposition alloy during HDDR-treatment also significant increase if hydrogen pressure increase from 0.2 to 0.5 MPa [30].

Because of this, the coercivity and anisotropy of the HDDR-treated powder of $Nd_2Fe_{14}B$ type hard magnetic alloy depends on hydrogen pressure (0.2-1.0 kgf/cm²) during decomposition stage, in particular coercivity increase with increasing of hydrogen pressure [31]. The hydrogen pressure effect on degree of magnetic anisotropy in $Nd_{12.5}Fe_{bal}Ga_{0.3}Nb_{0.2}B_{6.2}$ alloy during HDDR-treatment also was established, i.e. magnetic anisotropy was maximized when the sample was hydrogen disproportionated at 30 kPa (800°C-820°C) [32].

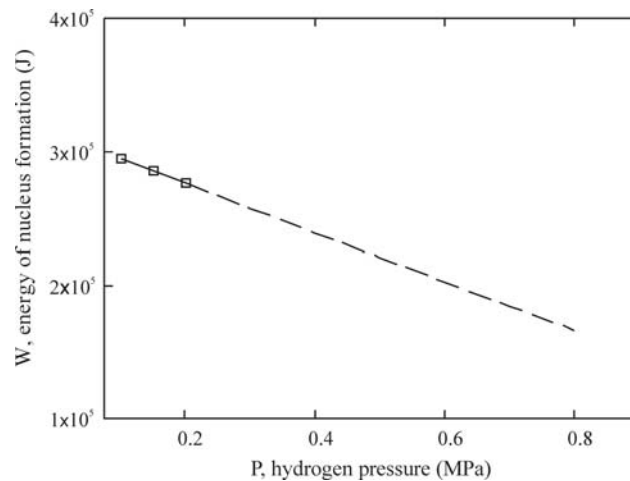


FIGURE 3. The critical nucleus energy of α -Fe phase formation W

dependence on hydrogen pressure P for direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy.

A similar effect was also discovered in $Nd_2Fe_{14}B$ -based sintered magnets where magnetic properties were the function of hydrogen pressure, temperature and treatment time [34].

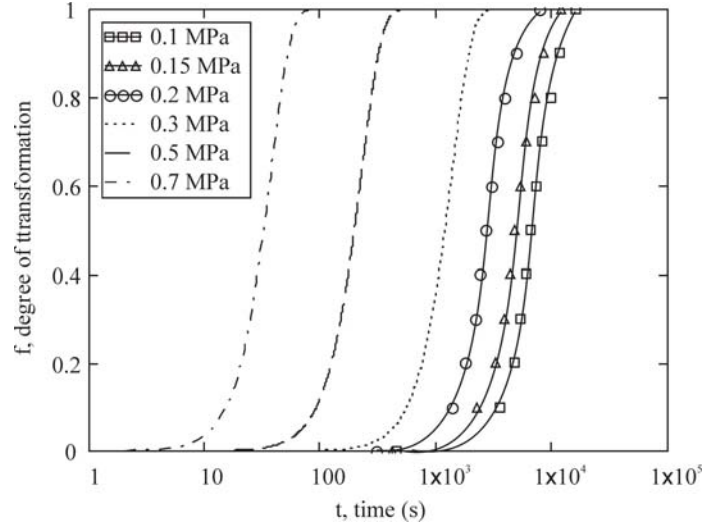


FIGURE 4. Kinetic curves (solid line) of direct hydrogen-induced phase transformation in $R_2Fe_{14}B$ hard magnetic alloy at $710^\circ C$ and hydrogen pressure of 0.10, 0.15 and 0.20 MPa (points – experimental data) and theoretical calculations for high pressure interval 0.3-0.7 MPa (dashed line).

For resin bonded magnets produced from $(Nd,Dy)_2Fe_{14}B$ type HDDR-treated powder it is established that remanence, coercivity and maximum energy product are function of the hydrogen pressure during the HDDR process [33].

Thus, in view of the above hydrogen pressure is not only important kinetic parameter determining of phase process developing, but also is very important parameter defining the properties of $R_2Fe_{14}B$ type permanent magnets produced from HDDR-treated powder of $R_2Fe_{14}B$ type alloys.

It is important for obtaining HDDR-treated powders with fine-grained structure, because as shown earlier non-compliance with the processing time leads to alloy abnormal grain growth processes and decreasing of permanent magnets magnetic properties [35-42], but treatment in accordance with kinetic features allows to prevent the abnormal grains growth processes [10,11].

From a practical viewpoint the obtained equation (10) and (11) can be used to calculate kinetics parameters (time pressure, temperature) of such transformations in $R_2Fe_{14}B$ alloy and obtain an optimal microstructure of $R_2Fe_{14}B$ alloy [1,2,4,38,40] for permanent magnets with high magnetic properties.

CONCLUSIONS

For evolution of hydrogen-induced direct phase transformations (decomposition in hydrogen atmosphere into rare-earth hydride RH_2 phase, α -Fe and Fe_2B) in hard magnetic $R_2Fe_{14}B$ ($R_{36.4}Fe_{62.45}B_{1.15}$, where R (Nd, Pr, Ce, 0.5% Dy)) type alloys kinetic model has been presented on the base of kinetic theory of the phase transformations in solid state. It is shown that the kinetic curve of hydrogen induced direct phase transformation in $R_2Fe_{14}B$ alloy in low temperatures range ($600-750^\circ C$) depending on hydrogen pressure can be described by the equation of the following type:

$$f(t) = 1 - \exp \left[- \frac{64\pi RT\gamma}{15h} \beta^3 D_o^{\frac{3}{2}} e^{-\frac{w+U+\frac{3}{2}Q}{RT}} t^n \right]$$

where R is the gas constant, T is the transformation temperature, h is the Planck constant, $\gamma=10^{-4}$ mol/m³, $\beta=10^{-3}$ is the kinetic parameter, $D_0=1$ mm²/s, Q is the activation energy of Fe atoms diffusion (250 kJ/mol), U is the activation energy of hydrogen atoms at transition of atoms through interface of RH₂ phases (-180 kJ/mol), W is the energy of critical nucleus of α -Fe phase formation which is function of hydrogen pressure – $W = -1.84 \cdot 10^5 \cdot P + 3.128 \cdot 10^5 P$ (P in MPa), t is the transformation time, $n=5/2$ is the kinetic constant. Therefore, with technological view point the obtained equation can be used for calculation of treatment time during HDDR-treatment of R₂Fe₁₄B type alloy for obtaining HDDR-treated powders with fine-grained structure and improved magnetic properties of magnets.

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