Hydriding properties of mechanically alloyed icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$
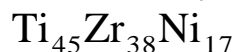
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Abstract

The interaction with hydrogen was investigated for mechanochemically synthesised icosahedral phase of the composition $Ti_{45}Zr_{38}Ni_{17}$. It was demonstrated that, unlike icosahedral phases of the same composition formed as a result of rapid quenching from melt or the annealing of alloys, hydriding of mechanochemically synthesized icosahedral phase at 503 K is not preceded by an induction period and starts at a maximal rate even when hydrogen pressure is less than 0.1 MPa. A diagram of the composition of icosahedral phase versus equilibrium hydrogen pressure (P–T–C diagram) at 503 K was experimentally obtained. Based on published data and the data obtained in our experiments, an assumption was made on the existence of two types of hydrogen positions in the icosahedral structure of the hydrogenated phase. The first type is more energetically bound hydrogen occupying the positions within the structure-forming icosahedral Bergman clusters. The concentration of such hydrogen is approximately corresponding to the relation $H/M = 1.1$. The second type is less bound hydrogen occupying the linkages between these clusters as solid solution. © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

Icosahedral phases, first discovered in 1984 [1], possess a unique crystallographic structure characterized by long range aperiodic order and crystallographically forbidden 5th order rotational symmetry. In crystallographic ordering degree they occupy an intermediate position between crystal and amorphous phases. Structural features can determine not only physical properties of icosahedral phases but also affect their reactivity. However, to date, only a few investigations have dealt with the chemical properties of icosahedral phases. One of pioneering works in this area was published by Kelton and co-workers [2–6] who discovered that icosahedral phases, formed in the system Ti–Zr–Ni as a result of melt-spinning or by the annealing of the alloy 45Ti + 38Zr + 17Ni, are capable of absorbing hydrogen with the formation of the solid solution of hydrogen in icosahedral structure. If the sample surface was not pretreated by plasma etching followed by palladium deposition [4,5], the icosahedral phase of the

composition $Ti_{45}Zr_{38}Ni_{17}$ absorbed hydrogen at a temperature 503–533 K and 2.7–4.0 MPa hydrogen pressure. Hydriding of these samples started only after a long induction period, varying from several hours to several days; the authors explained this phenomenon by presence of a protective oxide film on the sample surface preventing hydrogen chemisorption.

The icosahedral phases in some systems may be synthesized by mechanical alloying (MA), for example, in the systems Mg–Zn–Al, Mg–Cu–Al, Al–Cu–Mn [7–10]. It is also known that the phases obtained by mechanical alloying can possess increased reactivity. With that in mind, we studied the possibility of mechanochemical synthesis of icosahedral phase in the system Ti–Zr–Ni and its reactivity towards hydrogen.

2. Experimental

Mechanical alloying was carried out with a high-energy planetary centrifugal mill AGO-2 (acceleration about 400 m s^{-2}) with water-cooled vials. Stainless steel vials and balls of 5 mm in diameter were used. Ball mass to sample

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mass ratio was 20:1. To avoid oxidation, after loading the samples, the vials were evacuated and filled with dry argon gas.

Mechanical alloying of titanium, zirconium and nickel powder mixture results in the formation of rather uniform product layer on the balls and vial walls; it is split off the walls as scales about 300 μm thick and linear dimensions within several millimeters. Before X-ray analysis and hydriding, the samples were comminuted in an agate mortar. In order to minimize the size effect during hydriding, the fraction of particles between 60 and 200 μm was separated by sieve analysis. All the operations with the sample were carried out in air.

The interaction with hydrogen was investigated using a constant-volume reactor made of stainless steel. The amount of absorbed or evolved hydrogen was determined by following the pressure change in the reactor. The pressure was measured by piezoresistive transducer with overall precision better than 0.2%. Before hydriding, the reactor was evacuated and heated in dynamic vacuum about 5 Pa to the temperature required by the experiment. Temperature was maintained at an accuracy of ±1 K.

X-ray powder diffraction data were obtained with CuKα radiation.

3. Results and discussion

3.1. Synthesis of the icosahedral phase by mechanical alloying

Fig. 1 shows X-ray diffraction (XRD) patterns of the samples obtained after mechanical alloying of metal powder mixture (atomic ratio Ti:Zr:Ni=45:38:17) for different time. The pattern of the sample obtained by mechanical alloying for 0.5 h exhibits still rather clear though broadened peaks of the original metals; a broad halo is observed in the angle region $34<2\theta<41$ degree. Mechanical alloying for 1 h causes a practically complete disappearance of the initial phases in the XRD pattern. Only two very broad peaks are observed in the angle region corresponding to the positions of the two main peaks of the icosahedral phase. However, the intensity ratio of these peaks does not correspond to that of the reflections of icosahedral phase obtained by rapid quenching from melt. This is likely to be due to the residual amount of unreacted zirconium; its most intensive peak practically coincides with the reflection from the (100000) planes of the icosahedral phase. After 2 h of mechanical treatment, the appearance of XRD pattern, the positions of reflections and their intensity ratios correspond to the data reported in [2–6] for the icosahedral phase, but the peaks remain broadened substantially. The annealing of these samples in an inert atmosphere at 713 K for 4–5 h causes the narrowing of the peaks (pattern 4 in Fig. 1).
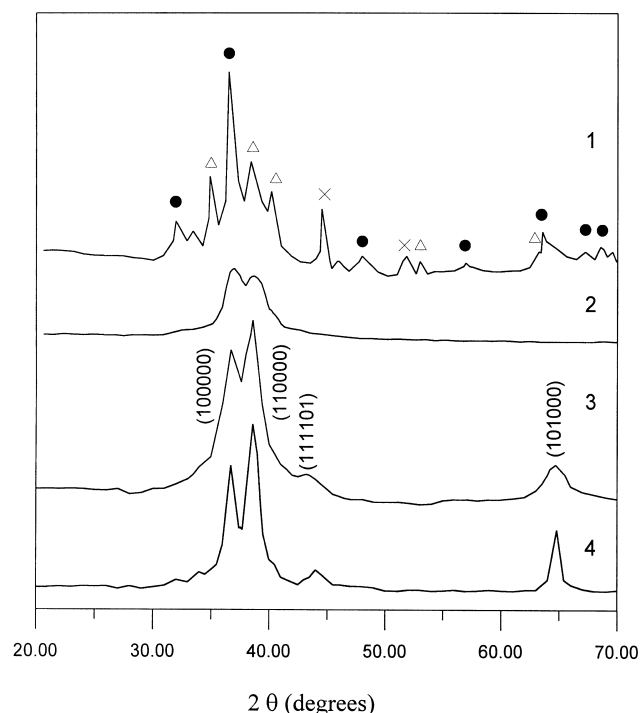
The sequence of changes in XRD patterns is similar to

Fig. 1. Diffraction patterns of the samples 45Ti+38Zr+17Ni obtained by mechanical alloying (MA) at the acceleration of 400 m s^{-2}. (1) MA for 0.5 h (●, Zr; △, Ti, X, Ni); (2) MA for 1 h; (3) MA for 2 h — the formation of icosahedral phase (4) annealing at 713 K in argon for 5 h after MA for 2 h.

that observed by us in the mechanochemical synthesis of the icosahedral phase in the system Mg–Zn–Al [9] (Fig. 2), although the times of mechanical treatment in these systems differ practically by an order of magnitude. The differences in the time of mechanical treatment necessary for the formation of icosahedral phases in these systems is likely to be explained by the fact that titanium, zirconium and nickel have much higher melting points than magnesium, zinc and aluminium. Due to this diffusion processes in the system Ti–Zr–Ni proceed much more slowly. Nevertheless, it can be concluded from the X-ray phase analysis data that the synthesis of icosahedral phases in these two systems proceeds via similar stages; it is probable that the same features will characterize mechanochemical synthesis of icosahedral phases in other systems as well.

The sequence of stages involved in the formation of icosahedral phases during mechanical alloying can be schematically represented as follows: as a rule, mutual dispersion of components and the formation of composites with large interface area take place at the initial stages of mechanical alloying as a result of repeated welding, fracturing and rewelding of a mixture of powdered metals (for example, see [11]). Due to the decrease of the size of coherent scattering blocks and the formation of defects, a decrease in the intensity and the broadening of the reflections of initial phases are observed in X-ray diffrac-
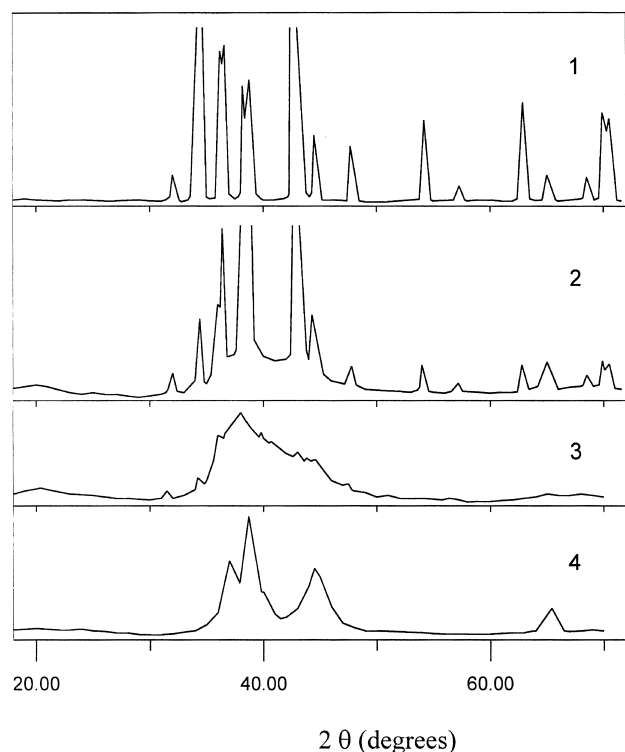
Fig. 2. Sequence of the diffraction patterns of the mechanically alloyed mixtures with the nominal composition of 3Mg:2Zn:3Al. (1) Original mixture of metal powders (2) MA for 6 min (3) MA for 12 min (4) MA for 20 min — the formation of icosahedral phase.

tion patterns. At the same time, the action of mechanical pulses and local heating causes mutual diffusion of the components in each other leading to the formation of icosahedral clusters that are the basic elements of icosahedral structure. Bergman type clusters can be used to describe the Ti–Zr–Ni system [12]. Bergman cluster consists of two concentric icosahedra, the radius of the outer one being twice that of the inner one. Metal atoms reside on the inner and outer verticals and outer face centers of these icosahedra.

Having formed, the icosahedral clusters can first build the amorphous structure, which manifests itself as a broad halo in XRD patterns. Further mechanical treatment intensifies mass transfer and leads to the crystallization of the non-translation, but more ordered and more thermodynamically stable, icosahedral phase. However, the reflections of this phase in XRD patterns are substantially broadened most likely due to the presence of defects in the icosahedral structure; annealing of the samples in an inert atmosphere causes the decrease of defect content and usually leads to narrowing of X-ray peaks [13].

3.2. Hydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$

The hydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ obtained by melt-spinning, usually accompanied by a long induction period and take place at 503–533 K and hydro-

gen pressure 2.7–4.0 MPa [2–4]. In our study we observed the hydriding (at 503 K) of the mechanically alloyed $Ti_{45}Zr_{38}Ni_{17}$ icosahedral phase starting without an induction period at the maximal rate even if the hydrogen pressure was below 0.1 MPa (Fig. 3). This may be due to the fact that the surface of the icosahedral phase samples prepared by mechanical alloying may contain more defects in comparison to the surface of the rapidly quenched samples. The surface of mechanically alloyed samples can contain insertions of the clusters of unreacted nickel or nickel oxide, the latter being easily reduced by hydrogen to form metal at 503 K. Nickel clusters are the centers of dissociative adsorption of hydrogen, which provides the absorption of hydrogen by the samples immediately after hydrogen is admitted into the reactor.

We came across this phenomenon for the first time when studying the hydriding of mechanical alloys of magnesium, formed at the initial stages of mechanical alloying of magnesium with d-elements (nickel, cobalt, iron) [14–16]. For mechanical alloys of magnesium with nickel, X-ray photoelectron spectroscopy (XPS) [17] showed that the surface oxide layer of the alloy particles was strongly disordered; the near-surface layer about 40 nm deep contains nickel clusters and isolated nickel atoms implanted into magnesium matrix. The disordering of the oxide layer makes these clusters and atoms of d-element accessible for hydrogen. Since d-elements are active catalysts of the dissociative adsorption of hydrogen, they promote the first hydriding of mechanical alloys, compared to usual alloys and mixtures of the same composition.

Recently, a rather large number of papers was published on the hydriding of intermetallic compounds synthesized by mechanical alloying or mechanical grinding (for exam-
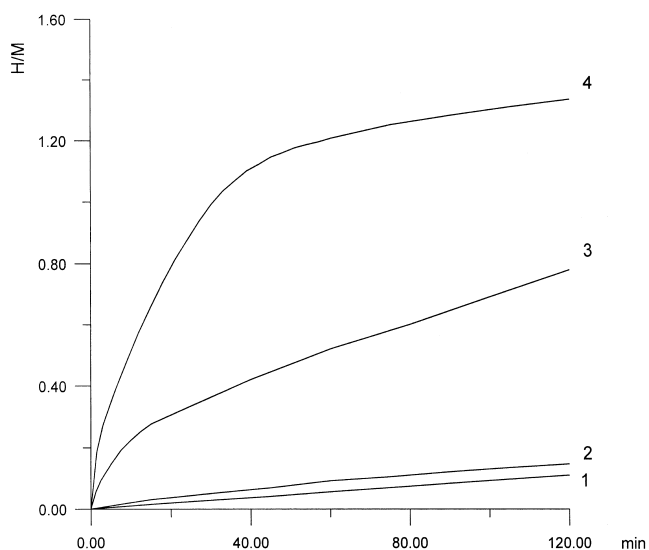

Fig. 3. Hydrogenation kinetics of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ at 503 K: (1,3) icosahedral phase obtained directly by mechanical alloying (2,4) icosahedral phase annealed after mechanical alloying (1,2) $P_{H_2} = 0.078$ MPa; (3,4) $P_{H_2} = 1.35$ MPa.

ple, see [18–21]). The authors noticed substantial increase in the rate of the first hydriding cycle, compared to similar materials obtained by conventional methods (alloying, annealing, etc.) and explain this fact by the formation of pure metal surface as a result of mechanical treatment, and dissociative adsorption of hydrogen taking place on this surface.

Fig. 3 shows the hydriding kinetic curves for the samples of icosahedral phase as mechanically alloyed, and the samples annealed in argon at 713 K for 5 h after mechanical alloying. The rate of hydriding of the annealed samples is higher than that of the samples after mechanical alloying. The differences can be due to two reasons; the degree of ordering of the icosahedral structure can be one reason. According to X-ray data (Fig. 1), the annealed samples are more ordered than the samples taken immediately after mechanical alloying. The diffusion of hydrogen in the ordered icosahedral structure may occur at a higher rate than in partially disordered structure. Another explanation can be the rupture of the surface oxide film during annealing, either because of cracking due to the differences in the thermal expansion coefficients of metal and oxide, or because of partial dissolution of the oxide. The rupture of the oxide film causes the formation of additional hydrogen adsorption centers and, as a consequence, the increase of hydriding rate. The fact that the activation of titanium and zirconium alloys occurs as a result of heating in vacuum or at low hydrogen pressure before hydriding is well known (see [22], for example). However, in our study, the annealed samples were exposed to the air before hydriding, which should have lead to the oxidation of the surface; yet the rate of their hydriding was higher than that of the samples after mechanical alloying.

The possibility to carry out hydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ at low hydrogen pressure and relatively low temperature (503 K) allowed us to build hydrogen absorption isotherm ($P-T-C$ diagram) for the icosahedral phase. Fig. 4 shows the data obtained with the preliminarily annealed samples of icosahedral phase. Equilibrium hydrogen pressure of the samples obtained directly by mechanical alloying practically coincides with the figures shown in the diagram. However, this can be the consequence of the fact that at 503 K the partially disordered icosahedral phase has enough time for the relaxation processes to take place within the long time intervals that are necessary for the equilibrium hydrogen pressure to be achieved. The differences, if any, in the $P-T-C$ diagrams of mechanically synthesized samples and the samples annealed after mechanical alloying are likely to be revealed experimentally only at, or close to, room temperature.

The maximum degree of sample saturation with hydrogen that was achieved in our experiments corresponded to the ratio $H/M = 1.6$ (H hydrogen, M metal atom), which agrees with the data, reported in [2–6]. The diffraction patterns of hydrogenated samples, similarly to those de-
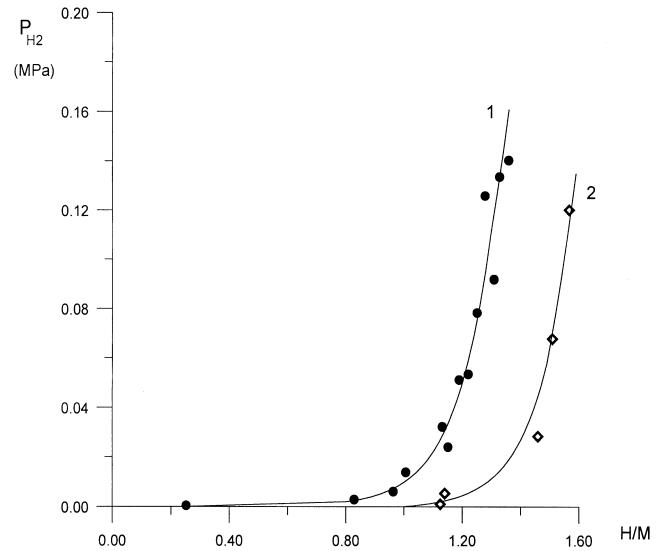

Fig. 4. Pressure–composition isotherm at 503 K for i-$Ti_{45}Zr_{38}Ni_{17}$ –H_2. (1) Hydrogenation (2) decomposition.

scribed in [2–6], exhibit the shifts of peaks to lower angles (diffraction pattern 5 in Fig. 5) which is characteristic of the formation of solid solutions. However, the sequence of changes in the diffraction patterns of the samples of icosahedral phase hydrogenated to different degrees of saturation with hydrogen, shown in Fig. 5, as well as the behavior of the pressure–composition isotherm (Fig. 4), allow us to conclude that the process of hydrogen absorp-
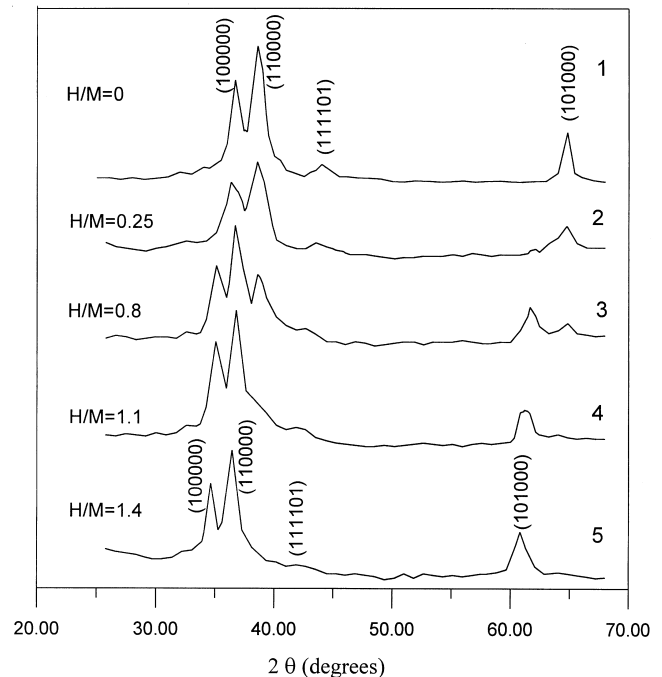

Fig. 5. Sequence of changes in X-ray diffraction patterns of samples obtained as a result of mechanical alloying, followed by annealing at 713 K, of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ after it has absorbed different amounts of hydrogen.

tion by the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ is characterized by the features not typical for the formation of true solutions. For the latter, the dependence of the equilibrium hydrogen pressure on composition should be described by the parabolic equation (Sivert's law) $P = k (H/M)^2$. In our case, only the right-hand part of the absorption isotherm obeys this law, starting approximately from the composition H/M = 1.1 (curve 1 in Fig. 4). At lower degree of saturation with hydrogen, the isotherm practically reaches the plateau pressure, which is characteristic of the systems forming chemical compounds (hydrides) [23].

The data of the X-ray phase analysis of the samples hydrogenated up to low degree of saturation with hydrogen can also prove that the hydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ is likely to involve a reversible chemical reaction and not only the dissolution of the gas in a solid. However, one cannot exclude that the formation of the true solid solution occurs at the initial stages of hydrogen absorption as it occurs in typical hydride-forming metal–hydrogen systems. At hydrogen saturation degree H/M = 0.25, the broadening of diffraction reflections is observed, along with insignificant shift of the maximums of the reflections of icosahedral phase to lower angles (diffraction pattern 2 in Fig. 5). This can be the evidence of the formation of low concentration solid solution of hydrogen. Further absorption of hydrogen in the sample results not in a gradual (as might be expected) shift of the reflections to lower angles but in the presence of both reflections of the initial and hydrogenated phases at the same time (diffraction pattern 3 in Fig. 5). The reflection (100000) of the initial phase and the reflection (110000) of the hydrogenated phase overlap. When hydrogen concentration in the icosahedral phase reaches H/M = 1.1, which corresponds to the region of inflection at the $P–T–C$ diagram, the reflections of the initial phase disappear practically completely, while the reflections of hydrogenated phase start to shift to the region of lower angles (diffraction pattern 4 in Fig. 5). At further saturation of the sample with hydrogen, for example up to the ratio H/M = 1.4, further shift of the reflections of hydrogenated phase into low angle region occurs (diffraction pattern 5 in Fig. 5). This is similar to the situation existing in typical metal–hydrogen systems forming hydrides, in which the plateau region at the $P–T–C$ diagram corresponds to the coexistence of the saturated solution with the composition $(H/M)_A$ and hydride phase with the composition $(H/M)_B$. Only after complete transformation of the whole sample into hydride, the equilibrium hydrogen pressure starts to increase. This is accompanied by the change of the composition of the hydride phase: a solid solution of hydrogen based on hydride is formed [23].

In the case when the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ is hydrogenated, the formation of a compound with an individual structure different from that of the initial phase does not occur. According to the data reported in [24], icosahedral structure is conserved at least till the degree of

saturation with hydrogen H/M = 1.88. However, the positions occupied by hydrogen in the icosahedral structure can be not equivalent crystallographically or energetically.

The authors of [24], who studied the hydrogenated icosahedral phases $Ti_{45}Zr_{38}Ni_{17}H_{188}$ and $Ti_{43}V_2Zr_{38}Ni_{17}H_{188}$ with nuclear magnetic resonance, used the model assuming that hydrogen atoms occupy some of the distorted tetrahedral sites (that are not closer together than 0.21 nm) within Bergman clusters, which consist of two concentric icosahedra. These authors performed theoretical calculations and revealed substantial discrepancy between experimental and theoretical ^{1}H second moment values. They concluded that the model does not take into account of substantial amount of hydrogen that probably occupies the linkages between Bergman clusters. An estimation shows that the experimental value of H/M = 1.88 is 1.7 times larger tan the theoretical value obtained from Bergman cluster model. Theoretical model taking into account only the hydrogen within Bergman's clusters predicts hydrogen concentration to be H/Me = 1.1. This may be not a chance coincidence with the inflection region of the absorption isotherm obtained by us for the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ (Fig. 4); this also corresponds to hydrogen concentration at which the reflections of the initial icosahedral phase disappear from X-ray diffraction patterns (pattern 4 in Fig. 5).

Thus, relying upon the data presented in [24] and taking into account our experimental data, we can assume that at least two kinds of hydrogen atoms exist in the icosahedral structure of hydrogenated phase $Ti_{45}Zr_{38}Ni_{17}$. The atoms of the first kind occupy the positions within Bergman clusters and is likely to be energetically bound to the neighboring metal atoms to a greater extent (according to the data of [25] hydrogen is located mainly in the vicinity of zirconium) than hydrogen atoms of the second type occupying the linkages between these clusters. This can also be confirmed by the time-of-flight neutron scattering study of hydrogen dynamics in icosahedral $Ti_{45}Zr_{38}Ni_{17}H_{150}$ quasicrystals performed in [26], where the authors conclude that some hydrogen atoms are present in this compound in chemically bound state.

Our data on the absorption isotherm can be interpreted as follows: it is possible that at the initial stages of hydrogen absorption, a true solid solution of hydrogen (of low concentration) is formed, similar to the case of classical hydride systems. Hydrogen is likely to be distributed randomly over the structure. When a definite hydrogen concentration is achieved, hydrogen starts to fill the distorted tetrahedral positions within Bergman clusters. A process similar to the crystallization of the hydride phase from supersaturated hydrogen solution takes place. The filling of these positions proceeds till hydrogen concentration H/M ≈ 1.1. Until this level is reached, we observe an inclined plateau at the absorption isotherm, and the XRD peaks of two phases (initial icosahedral and hydrogenated icosahedral). Only after hydrogen occupies all the

positions crystallographically allowed inside Bergman clusters, it starts to fill the linkage between Bergman clusters. Since this moment, the equilibrium hydrogen pressure starts to increase obeying the Siverts' law; XRD patterns exhibit further shifts of the diffraction reflections to the lower angles.

Hydrogen desorption isotherm (curve 2 in Fig. 4) obtained as a result of dehydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}H_{160}$ at 503 K is the evidence that, like in most hydride-forming systems, hysteresis phenomenon exists in the system $Ti_{45}Zr_{38}Ni_{17}-H_2$. The equilibrium hydrogen pressure achieved during dehydriding of the samples is somewhat lower than that observed during hydriding. Dehydriding causes a shift of the XRD reflections of the icosahedral phase to higher angles. However, at 503 K the hydrogen absorbed by the sample is not completely evolved. The concentration of hydrogen remaining bound at this temperature was $H/M \approx 1.1$. This can be one more confirmation in favor of the fact that a part of hydrogen (its concentration corresponding to the theoretical hydrogen concentration occupying distorted tetrahedral positions within Bergman clusters) in the icosahedral $Ti_{45}Zr_{38}Ni_{17}H_x$ is in more energetically bound state and requires higher temperature for decomposition.

4. Conclusion

Mechanical alloying in the system Ti–Zr–Ni leads to the formation of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$. The MA synthesized $Ti_{45}Zr_{38}Ni_{17}$ has an increased reactivity with hydrogen, compared to similar phases obtained by melt-spinning or the annealing of alloys.

The hydriding of the icosahedral phase $Ti_{45}Zr_{38}Ni_{17}$ at low hydrogen pressure allowed to determine the equilibrium hydrogen pressure for this phase and to build up a P–T–C diagram.

From the analysis of the P–T–C diagram and X-ray diffraction data it has been concluded that the process of hydrogen absorption by the icosahedral phase is similar to the formation of chemical compounds, i.e. hydrides.

In order to explain this phenomenon and also in agreement with the literature data, we assumed that at least two kinds of hydrogen exist in the structure of the hydrogenated icosahedral phase. These are energetically more bound hydrogen that occupies the distorted tetrahedral positions within the structure-forming icosahedral Bergman clusters, and energetically less bound hydrogen occupying the linkages between these clusters as a solid solution. The concentration of more bound hydrogen corresponds approximately to the ratio $H/M = 1.1$, which coincides with the concentration of hydrogen occupying the positions within Bergman clusters, calculated theoretically in [24].

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