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STUDY OF NANOCRYSTALLINE SUBSTANCES WITH SORBED HYDROGEN BY NEUTRON SCATTERING

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Исследование нанокристаллических веществ с сорбированным
водородом с помощью рассеяния нейтронов

В работе П. Халла и др. [1] показано, что нанокристаллические сплавы FeTi
более предпочтительны для хранения водорода по сравнению со сплавами, при-
готовленными по обычной технологии. Дифракционные и вибрационные спек-
тры нанокристаллических образцов с сорбированным водородом $(FeTiMg)Hx$,
$(MgTi)Hx$, $(MgTiPd)Hx$, $(FeTiMn)Hx$, $[(FeTiMn)Mg]Hx$ получены с помощью рас-
сеяния нейтронов. Наблюдались особенности кристаллической структуры этих
образцов. Выделены фононные и локальные вибрационные моды сорбирован-
ного водорода, и определены их энергии. Показано, что энергии водородных
мод в многокомпонентных нанокристаллических структурах с сорбированным
водородом не описываются просто энергиями водородных мод, наблюдавшимися
в различных однокомпонентных гидридах металлов.

Работа выполнена в Лаборатории нейтронной физики им. И. М. Франка
ОИЯИ.

Hall P. et al.   Е14-2007-65

Study of Nanocrystalline Substances with Sorbed Hydrogen
by Neutron Scattering

Earlier P. Hall et al. [1] have shown that FeTi nanocrystalline alloys are more
preferable for hydrogen storage in comparison with those prepared by usual technol-
ogy. The diffraction and vibration spectra from nanocrystalline samples with sorbed
hydrogen of $(FeTiMg)Hx$, $(MgTi)Hx$, $(MgTiPd)Hx$, $(FeTiMn)Hx$, $[(FeTiMn)Mg]Hx$
are obtained by means of neutron scattering. The crystal structure peculiarities of
the samples are observed. The phonon and local vibrational modes of sorbed hy-
drogen are selected and their energies are determined. It is shown that the energies
of hydrogen modes in multicomponent nanocrystalline structures with sorbed hydro-
gen are not described simply by energies of hydrogen modes observed in different
single-component metal hydrides.

The investigation has been performed at the Frank Laboratory of Neutron Physics,
JINR.

INTRODUCTION

The realization of hydrogen as a fuel is determined by the level of the development of a technology for effective hydrogen storage. For practical applications, metal hydrides must be cheap, have adequate hydrogen uptakes at reasonable temperatures and have long lifetimes. Considerations of many metal alloys investigated previously have led to selection of relatively cheap FeTi and Mg based systems.

The disadvantage of the most metal hydride systems that can be developed for practical applications, is that they need an extended activation process at high temperatures and pressures before the materials will take up useful amounts of hydrogen. FeTi based alloys have been studied for a long time and have been largely ignored for development as practical hydrogen storage systems because of this problem. However, P. J. Hall et al. [1] have shown that FeTi alloys when produced in a nanocrystalline form by vapour deposition avoid this problem. High-pressure differential scanning calorimetry shows that these materials have good hydrogen cycling characteristics. They also take up and release hydrogen at reasonable temperatures.

It is also known that various hydrogen hydrides content bubbles in which significant amount of hydrogen is presented at high pressure. In fact, gas bubbles within metals are a fairly common occurrence and special care needs to be taken if one needs to avoid them. What appeared to be happening is that the high-pressure hydrogen bubbles appeared to be stabilized by the surrounding non-porous metal hydride. In the context of developing hydrogen storage media this is an interesting development because it opens the possibility of deliberately engineering gas bubbles in metals to increase their storage capacity.

However, before such a development can be contemplated, more information about the state of hydrogen within these metals is needed. Neutron diffraction and inelastic neutron scattering are ideal techniques in this context.

1. EXPERIMENTAL DATA

The investigation of different substances with nanocrystalline structure with sorbed hydrogen by means of neutron powder diffraction and inelastic incoherent neutron scattering are carried out at the IBR-2 impulse reactor by means of the time-of-flight method at the DN-2 diffractometer at room temperature and at the NERA-PR spectrometer at 20 K (FLNP JINR, Dubna, Russia) and at the IN1 BeF spectrometer of steady reactor (ILL, Grenoble, France) in the temperature region from 5 to 50 K.

1.1. The Study of [(FeTi)Me]Hx (Me = Mg, Mn) Nanocrystalline Materials with Sorbed Hydrogen. The FeTi nanocrystalline materials with sorbed

hydrogen are studied with different alloying ingredients as Mg and Mn. The neutron powder diffraction (NPD) spectra for (FeTiMg)Hx and (FeTiMn)Hx at 20 K and in the range of interplane distances from 1.5 to 8 Å are presented in Fig. 1, *a, b*. The presented NPD spectra have common peculiarities in view of broad peaks with $d = 2.976$, and 1.718 Å for (FeTiMg)Hx and with $d = 2.983$, and 1.730 Å for (FeTiMn)Hx.
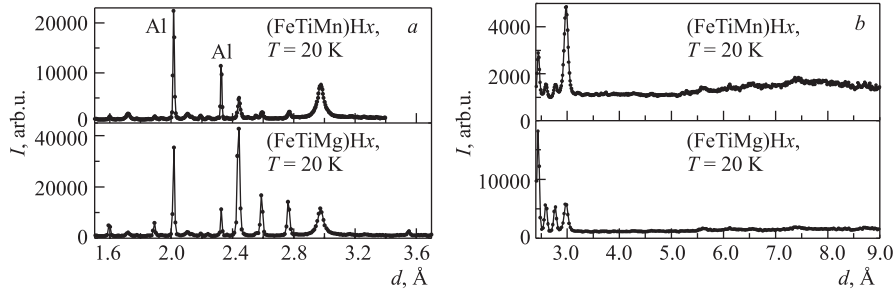

Fig. 1. Neutron powder diffraction spectra of (FeTiMg)Hx and (FeTiMn)Hx at 20 K: *a*) $2\Theta = 143.2°$ and *b*) $2\Theta = 45°$

The inelastic incoherent neutron scattering (IINS) spectra, obtained for (FeTiMg)Hx and (FeTiMn)Hx nanocrystalline samples at the NERA-PR spectrometer at 20 K, are transformed in generalized vibrational density of states $G(E)$ spectra in the energy range up to 200 meV, which are presented in Fig. 2.
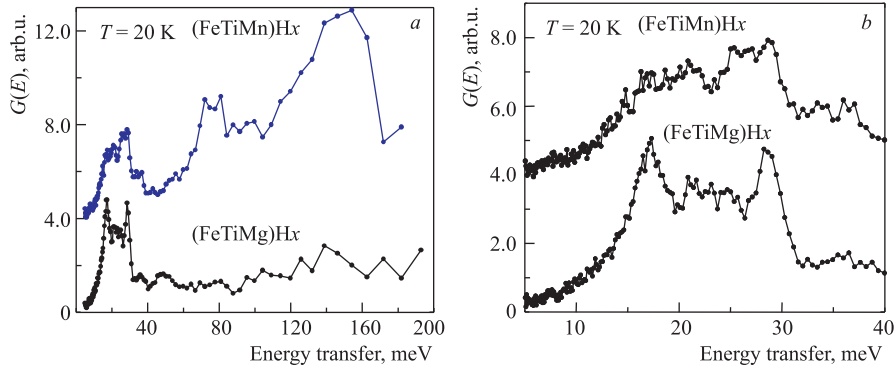

Fig. 2. The generalization vibrational density of states spectra of (FeTiMg)Hx and (FeTiMn)Hx measured at the NERA-PR spectrometer at the temperature of 20 K: *a*) energy transfer region up to 200 meV, *b*) energy transfer region up to 35 meV

The NERA-PR spectrometer gives the possibility to obtain inelastic incoherent neutron scattering spectra for energy transfer more than 0.6 up to 400 meV, but

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the density of neutron beam of the IBR-2 reactor is not enough to obtain IINS spectra in large energy transfer region with good statistics.

Thus by using of IINS the information about phonon density of states and vibrational optical or local energies of hydrogen modes are obtained. The $G(E)$ spectrum for (FeTiMg)Hx nanocrystalline sample shows the presence of phonon hydrogen modes, but local hydrogen modes in this samples are not observed. The $G(E)$ spectrum for (FeTiMn)Hx nanocrystalline sample shows the presence of phonon hydrogen modes and local hydrogen modes in energy transfer region up to 200 meV (see Fig. 2, a). The phonon hydrogen modes of (FeTiMg)Hx and (FeTiMn)Hx in energy transfer range up to 35 meV are presented in detail in (Fig. 2, b). The comparison of the $G(E)$ spectra of phonon hydrogen modes for (FeTiMg)Hx and (FeTiMn)Hx shows that phonon density of state for (FeTiMg)Hx nanocrystalline sample has more fine structure than for (FeTiMn)Hx nanocrystalline sample. The selected maxima of peaks can suited to values of energies on the boundaries of Brillouin zone. Such a fine shape of phonon density of states for (FeTiMg)Hx can be explained by the ordered distribution of hydrogen atoms within unit cells of grains. The shapes of phonon density of states for (FeTiMn)Hx nanocrystalline sample are presented by width maxima showing that hydrogen atoms can occupy the lattice sites inside of grains and also over surfaces of grains. In this case the complex shape of the $G(E)$ spectrum for (FeTiMn)Hx consists from vibrational modes of ordered hydrogen atoms inside of grains and from disordered hydrogen atoms located inside of grain boundaries.

The $G(E)$ spectra of (FeTiMg)Hx and (FeTiMn)Hx nanocrystalline samples are analyzed and the energies of selected hydrogen modes are presented in Table 1.

1.2. The Study of Nanocrystalline Materials with Sorbed Hydrogen Based on (MgTi)Hx. The NPD spectra of $(Mg_{0.90}Ti_{0.10})$Hx and $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ nanocrystalline materials with sorbed hydrogen, measured at the NERA-PR spectrometer at 20 K in the range of the interplane distances d from 1.5 to 8 Å, are presented in Fig. 3. The NPD spectrum of $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ nanocrystalline sample does not have significant difference from the NPD spectrum of the $(Mg_{0.90}Ti_{0.10})$Hx nanocrystalline sample.

The IINS spectra of these nanocrystalline materials with sorbed hydrogen are measured also at the NERA-PR spectrometer at 20 K. The $G(E)$ spectra for the $(Mg_{0.90}Ti_{0.10})$Hx and $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ nanocrystalline materials are presented in Fig. 4, a in energy transfer range up to 200 meV. The $G(E)$ spectra for phonon hydrogen modes of these nanocrystalline samples are presented in Fig. 4, b in the energy transfer region up to 35 meV.

The comparison of the $G(E)$ spectra of the $(Mg_{0.90}Ti_{0.10})$Hx and $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ nanocrystalline samples shows that addition of 1% Pd does not change significantly these spectra similar to the NPD spectra. The $G(E)$ spectra of these nanocrystalline samples have phonon hydrogen modes and local modes of the first and second harmonics. The phonon hydrogen modes have

some fine structure. Possibly the addition of 1% Pd has an effect on the phonon hydrogen modes changing the shape of the $G(E)$ spectrum in this part of energy transfer. So if the shape of the $G(E)$ spectrum in energy transfer region
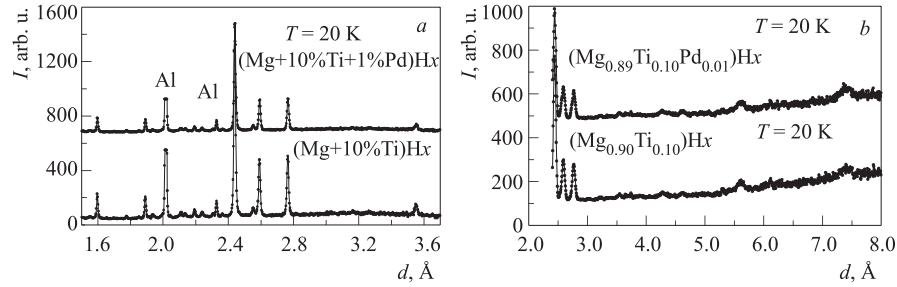

Fig. 3. Neutron powder diffraction spectra of $(Mg_{0.90}Ti_{0.10})Hx$ and $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ at 20 K: *a*) $2\Theta = 143.2°$ and *b*) $2\Theta = 45°$
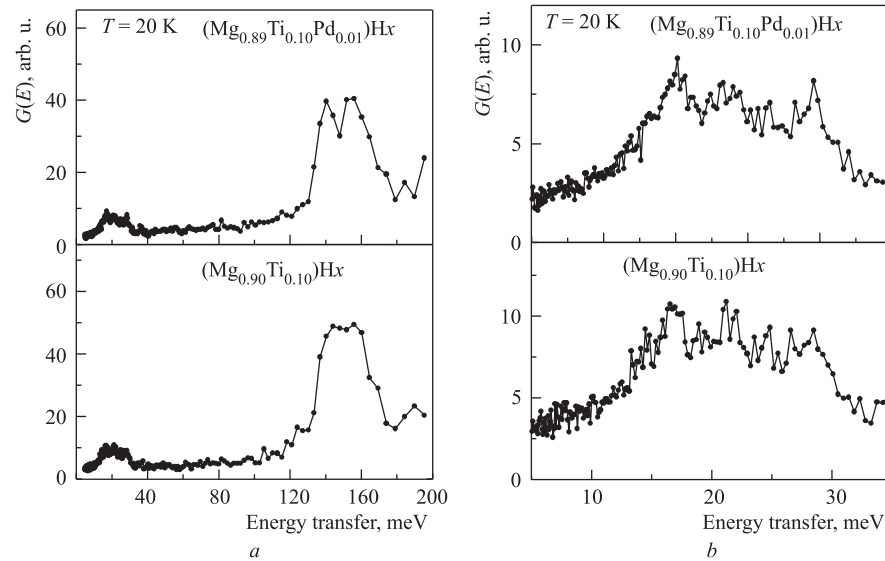
Fig. 4. The $G(E)$ spectra of $(Mg_{0.90}Ti_{0.10})Hx$ and $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ (NERA-PR spectrometer, at the temperature of 20 K: *a*) energy region up to 200 meV, *b*) energy region of phonon modes

of phonon modes of $(Mg_{0.90}Ti_{0.10})Hx$ can be presented by the contribution from 5 modes, then similar shape of the $G(E)$ spectrum for $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ is

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presented only by 3 modes. This change appears for account of peak broadening within part of the $G(E)$ spectrum of phonon hydrogen modes. The energies of selected hydrogen modes for these nanocrystalline samples are determined and presented in Table 1.

1.3. The Measurements of Nanocrystalline Materials with Sorbed Hydrogen Studied at the IN1 BeF Spectrometer. The nanocrystalline materials with sorbed hydrogen S1–$(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$, S2–$(FeTiMn)Hx$, S3–$[(FeTiMn):Mg]Hx$ (weight relation), S4–$(Mg_{0.895}Ti_{0.10}PCA_{0.005})Hx$, S5–$[(FeTiMn)_{0.6}:Mg_{1.4}]Hx$ (weight relation) are measured at the IN1 BeF spectrometer at different temperatures. The samples S1 and S2 are measured at 5 and 50 K, respectively, and S3–S5 samples are measured at 10 K, and their IINS spectra $(I(E))$ are presented in Fig. 5. The measurements of the IINS spectra of these samples are carried out from 27 to 200 meV.
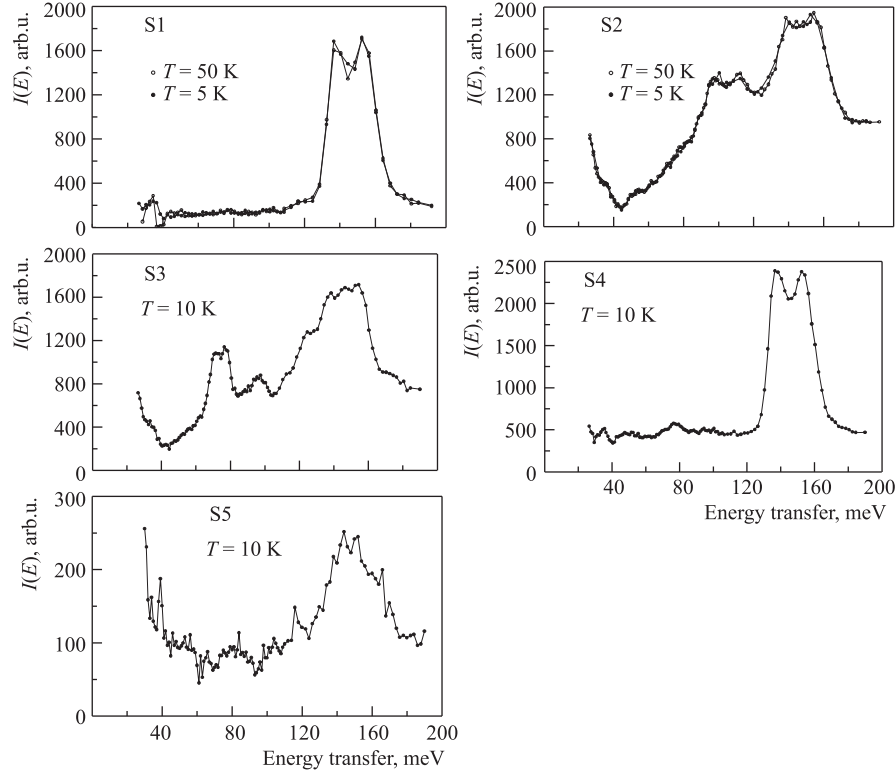

Fig. 5. The measured spectra of nanocrystalline samples at the IN1 BeF spectrometer: S1–$(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$, S2–$(FeTiMn)Hx$, S3–$[(FeTiMn):Mg]Hx$ (weight relation), S4–$(Mg_{0.895}Ti_{0.10}PCA_{0.005})Hx$, S5–$[(FeTiMn)_{0.6}:Mg_{1.4}]Hx$ (weight relation)

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Usually the IN1 BeF spectrometer gives the possibility to carry out the measurements of the inelastic incoherent neutron scattering for the energy transfer of more than 30 meV. The spectrometer is suited to obtain the information about vibrational optical or local energies of hydrogen modes.

The result of the analysis of obtained spectra for generalized vibrational density of states of above-mentioned nanocrystalline materials with sorbed hydrogen is presented in Table 1. Table 1 contains selected hydrogen phonon and local modes and their corresponding energies.

Table 1. The energies of hydrogen modes determined in nanocrystalline substances (energy in meV)

Sample	Spectrometer	Lattice bands	Local bands
FeTi	NERA-PR		
$(FeTiMn)Hx$	NERA-PR	18.3, 27.0	72.2, 79.6, 142.2, 158.5
$(FeTiMg)Hx$	NERA-PR	16.9, 21.0, 22.6, 24.7, 28.5	
$(Mg_{0.90}Ti_{0.10})Hx$	NERA-PR	16.6, 19.5, 21.5, 24.6, 28.0	142.5, 157.0
$(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$	NERA-PR	16.7, 21.9, 28.4	139.6, 155.9
$S1–(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$	IN1 BeF		137.4, 152.7
$S2–(FeTiMn)Hx$	IN1 BeF		99.8, 115.6, 140.3, 156.7
$S3–[(FeTiMn):Mg]Hx$	IN1 BeF		58.1, 72.8, 78.3, 94.6, 121.3, 141.4, 155.9, 171.8
$S4–(Mg_{0.895}Ti_{0.10}PCA_{0.005})Hx$	IN1 BeF		77.7, 137.2, 152.2
$S5–[(FeTiMn)_{0.6}:Mg_{1.4}]Hx$	IN1 BeF		144.9

2. DISCUSSION

It is known that nanocrystalline materials can be treated as polycrystals with grain sizes of a few nanometers, namely, approximately, 2–50 nm [2] or even ~2–30 nm [3]. Such small grain sizes significantly increase the fraction of the atoms which occupy grain boundaries and this new situation leads to new physical properties [4]. This new situation is accompanied for hydrogen atoms by new possibilities to locate inside of nanocrystalline materials. Now hydrogen

atoms can occupy as tetra- and octa-pores inside of unit cell as well as the sites of originated due to heterogeneities (pores, internal macroscopic density fluctuations and surface roughness) [4]. As a result of these new possibilities for hydrogen atoms to occupy different sites in nanocrystalline materials their generalized vibrational density of states can turn more complex in comparison with that of coarse-grained polycrystalline materials.

The influence of nanocrystalline structure on generalized vibrational density of states can be traced to different causes. First, the influence of small grain size can lead to the change of energies of vibrations of hydrogen atoms occupied tetra- and octa-pores inside of unit cell or other crystallographically independent sites. The cause of such a change can be due to the deformational potential which arises under the influence of surface atoms of a grain.

Second, one can expect the possible influence on $G(E)$ nanocrystalline material from vibrations of hydrogen atoms which occupy sites over the surface of the grain boundary. Grain boundary can coincide with different directions of crystal planes that can lead to different vibrational energies for hydrogen atoms. There can be other additional causes for the change of vibrational energies of hydrogen atoms. The contribution of all these additional changes to common generalized vibrational density of states leads to the different distribution than the original $G(E)$ for coarse-grained polycrystalline material.

From general consideration of the problem of hydrogen vibration in nanocrystalline materials one can expect to observe hydrogen modes of low and high energies. Indeed, for studied nanocrystalline materials (FeTiMg)Hx, (FeTiMn)Hx, (MgTi)Hx and (MgTiPd)Hx these energy regions are observed. The high-energy vibrations of hydrogen atoms can be attributed to hydrogen atoms which locate lattice sites inside of grains and also over boundaries between grains. The low-energy vibrations can be explained as hydrogen vibrations which take place in vibrations of the lattice of grains or grain boundary. The hydrogen atoms can be ordered or disordered over grain boundary and the grain boundary can coincides with some well-defined crystal faces. In this case $G(E)$ will be presented by maxima which are correspond to energies on the boundaries of Brillouin zones. Such a case is observed in $G(E)$ spectrum of (FeTiMg)Hx for which one can suppose that hydrogen atoms are ordered in this nanocrystalline substance (see Fig. 2, b). On the other hand, low energy part of $G(E)$ spectrum for (FeTiMn)Hx is smearing in comparison with that of (FeTiMg)Hx for the account of possible disorder of hydrogen atoms.

Thus obtained $G(E)$ spectra for studied nanocrystalline materials (FeTiMg)Hx, (FeTiMn)Hx, (MgTi)Hx and (MgTiPd)Hx have interesting peculiarities which give the information about structure and dynamics of hydrogen atoms. So the analysis of these triple nanocrystalline alloys indicates the observation of the first and second harmonics as 72.2, 79.6 and 142.2, 158.5 meV in (FeTiMn)Hx, 142.5, 157.0 meV in (Mg$_{0.90}$Ti$_{0.10}$)Hx, 139.6, 155.9 meV in (Mg$_{0.89}$Ti$_{0.10}$Pd$_{0.01}$)Hx,

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72.8, 78.3 and 141.4, 155.9 meV in S3–[(FeTiMn):Mg]Hx. It is interesting to notice, that quaternary S3–[(FeTiMn):Mg]Hx nanocrystalline alloy contains additional hydrogen modes with energies 58.1, 94.6, 121.3, 171.8 meV outside the first and second harmonics as in the triple (FeTiMn)Hx alloy. These points on different additional potential wells which are formed for embedded hydrogen atoms in quaternary alloy in comparison with triple appearing possibly as in grains or within boundaries between grains.

However, it is interesting to continue this investigation with multicomponent alloys which must be prepared as coarse grain materials with hydrogen atoms in comparison with investigated nanocrystalline materials with sorbed hydrogen. Then the probability of the influence of boundaries between grains will be limited up to minimum.

The observed low energies of phonon modes and high energies of vibrational local modes are known in general for hydrogen atoms embedded in pure elements. Table 2 contains the hydrogen modes in coarse grains of nanocrystalline materials of clear elements in comparison with energies of phonon and local modes hydrogen with investigated nanocrystalline materials with sorbed hydrogen.

The analysis of observed energies of hydrogen modes, obtained by inelastic incoherent neutron scattering, in coarse grains of clear metallic elements doped by hydrogen and investigated nanocrystalline materials sorbed hydrogen shows, that there are several close values of energies of hydrogen modes. So there are hydrogen modes with energies 135 and 158 meV in $MgH_{0.5}$ with structure of rutile [5] and there are hydrogen modes with energies 135 and 146 meV in $TiH_{1.96}$ [13]. On the other hand, there are hydrogen modes, measured at the NERA-PR spectrometer, in nanocrystalline alloys with sorbed hydrogen (FeTiMn)Hx with energies 142.2 and 158.5 meV, in $(Mg_{0.90}Ti_{0.10})$Hx with energies 142.5 and 157.0 meV and in $(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ with energies 139.6 and 155.9 meV. Hydrogen modes, determined at the IN1 BeF spectrometer are observed among investigated nanocrystalline alloys with sorbed hydrogen in S1–$(Mg_{0.89}Ti_{0.10}Pd_{0.01})Hx$ with energies 137.4 and 152.7 meV, in S2–(FeTiMn)Hx with energies 140.3 and 156.7 meV, in S3–[(FeTiMn):Mg]Hx with energies 141.4 and 155.9 meV, in S4–$(Mg_{0.895}Ti_{0.10}PCA_{0.005})Hx$ with energies 137.2 and 152.2 meV and in S5–$[(FeTiMn)_{0.6}:Mg_{1.4}]Hx$ with energy 144.9 meV. These selected energy values for hydrogen modes have insignificant discrepancies, which reflect the peculiarities of interactions embedded hydrogen atoms with surrounding their neighboring atoms.

There are also additional hydrogen modes with energies, which are not observed in rutil-$MgH_{0.5}$ and $TiH_{1.96}$, as, for example, energies 99.8 and 115.6 meV in S2–(FeTiMn)Hx, 58.1, 94.6, 121.3 and 171.8 meV in S3–[(FeTiMn):Mg]Hx. There are pairs of energies as 72.2 and 79.8 in (FeTiMn)Hx, 72.8 and 78.3 meV in S3–[(FeTiMn):Mg]Hx, which are close only to pairs of energies in rutil-$MgH_{0.5}$ as 80 and 95 meV.

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Table 2. The energies of hydrogen modes in different metals (energy in meV)

Metal hydride	Energy, meV		Ref.
	Lattice band	Local band	
rutile $MgH_{0.5}$		80, 95, 135, 158	[5]
fcc γ-$MnH_{0.41}$		111, 222, 333	[6]
$\alpha-MnH_{0.073}$		73, 105, 123	[7]
Nano Fe	Band 20–30		[8]
Nano- ^{57}Fe	35		[9]
Powder PdH		55.8, 58.0, 78.5	[10]
$PdH_{0.99\pm0.03}$, $\psi = 0$:	13.7, 22.5	55.8, 58.0, 78.5, 116.2, 148.7, 252.8, 363.8, 474.5	[11]
$PdH_{0.99\pm0.03}$, $\psi = 45^o$:		112, 113.5, 116.8, 135.3, ~173, 200.2, 290	[12]
$TiH_{1.96}$	0–40	135, 146,	[13]
n-PdH		50–80	[2]
dhcp-FeH		105	[14]
ε'-FeH		103, 206, 309	[15]
$(Fe_{25}Cr_{20}Ni)H_{0.43}$		122	[16]
$(Fe_{25}Cr_{20}Ni)H_{0.92}$		113	

Thus investigated triple nanocrystalline samples with sorbed hydrogen are prepared on the basis of Fe, Mg, Ti, Mn and Pd. The investigation of these triple nanocrystallines by the IINS shows that there are observed hydrogen phonon and local modes which earlier were observed in coarse grain materials doped hydrogen, as rutile $MgH_{0.5}$ and $TiH_{1.96}$. The energies of hydrogen modes observed in nanocrystalline alloys correspond with insignificant differences to energies observed in coarse grains of clear elements and also to additional modes with different energies.

It is of some interest to compare samples S2, S3 and S5 in which the concentrations of components are changed. So the three-component nanocrystalline sample S2, composed from Fe–Ti–Mn with sorbed H, significantly changes the set of local hydrogen modes with energies 99.8, 115.6, 140.3 and 156.7 meV with the addition of Mg in the proportion [(FeTiMn):Mg]Hx to the set of local hydrogen modes with energies 58.1, 72.8 and 78.3, 94.6, 121.3, 141.4 and 155.9 meV of sample S3. Possibly energies of the pairs (99.8 and 115.6) and (140.3 and 156.7) meV for S2 sample are congenial to energies of pairs (94.6 and 121.3) and (141.4 and 155.9) meV for S3 sample because some clusters are approximately conserved under alloying with Mg. On the other hand, the appear

of new hydrogen modes with energies 58.1 and 72.8 in S3 sample points out the formation of new clusters. However, in S5 sample with concentration composition [(FeTiMn)0.6:Mg1.4]Hx the set of hydrogen modes is significantly changed and presented only the mode with energy 144.9 meV, pointing out significant reconstruction of nanocrystalline structure.

CONCLUSIONS

The studies of nanocrystalline materials (FeTiMg)Hx, (FeTiMn)Hx, (MgTi)Hx, (MgTiPd)Hx and (FeTiMnMg)Hx by means of powder neutron diffraction and inelastic incoherent neutron scattering gave the possibility to obtain the following results. It is shown that diffraction spectra have some Bragg reflections with more large widths in comparison with others, confirming the formation of nanocrystalline structures. The above-mentioned nanocrystalline compounds with embedded hydrogen manifest hydrogen phonon modes in low energy region which can represent the order or disorder of hydrogen atoms. If the $G(E)$ spectra are presented by narrow peaks then hydrogen atoms are ordered within grains or over well-defined crystal faces of grain boundaries. On the other hand, if the $G(E)$ spectra are presented by smearing peaks then hydrogen atoms are disordered within grains and grain boundaries. The studied many-component nanocrystalline compounds with embedded hydrogen manifest hydrogen local modes of high energy which are observed in unicomponent compounds and besides additional modes. Thus determined energies of selected hydrogen modes in studied many-component nanocrystalline samples could not be explained for account of hydrogen modes of unicomponent compound which compose many-component nanocrystalline compounds.

There is one question which is not described in the main description: it is the problem of bubbles in the nanocrystalline samples. If bubbles are formed indeed within nanocrystalline alloys and are filled with hydrogen then below 14 K it transforms to solid state. It is known that hydrogen in solid state has mode at ~ 14.7 meV [17], which can be observed by the inelastic incoherent neutron scattering. Our measurements are carried out at the NERA-PR spectrometer at 20 K and in energy transfer region beginning from ~ 1.5 meV and at the IN1 BeF spectrometer at 5 and 50 K in energy transfer region beginning from ~ 27 meV. The search of hydrogen filled bubbles in nanocrystalline alloys is interesting to carry out at necessary conditions.

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