Hydriding of TiMo Alloys at High Hydrogen Pressures

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Abstract—We studied the interaction of $Ti_{0.40}Mo_{0.60}$ and $Ti_{0.34}Mo_{0.66}$ alloys with hydrogen and obtained hydrogen desorption isotherms at pressures of up to 250 MPa. At high hydrogen pressures, we observed the formation of $Ti_{0.40}Mo_{0.60}H_{1.1}$ and $Ti_{0.34}Mo_{0.66}H_{0.8}$ hydride phases. According to X-ray diffraction data, the hydrides consisted of phases with a body-centered cubic and face-centered cubic (CaF_2 structure) lattices. The structure of the deuteride based on the $Ti_{0.40}Mo_{0.60}$ alloy was studied by neutron diffraction. We identified the sites occupied by deuterium atoms and determined their occupancies.

INTRODUCTION

Intermetallic and metal alloy hydrides are promising materials for compact and safe hydrogen storage [1, 2]. Data in the literature provide detailed insight into the interaction of hydrogen with titanium- and molybdenum-based alloys at low pressures (10 MPa or lower) [3–8]. For example, Antonova and Chernogorenko [3] studied the interaction of Ti–Mo alloys in the composition range 7 to 60 wt % molybdenum (5 to 46 at % Mo) with hydrogen at a pressure of 0.1 MPa. According to X-ray diffraction data, the resultant hydrides had a face-centered cubic (FCC) lattice (CaF_2 structure). It has been shown that increasing the molybdenum content of the alloys reduces the hydrogen content of the hydrides and the hydrogen release temperature.

Wang et al. [4] investigated the interaction of a mixture of hydrogen and deuterium with Ti–Mo alloys in the composition range 3 to 40 at % molybdenum. Their results demonstrate that hydrogen and deuterium react with the alloys in different ways, suggesting that such alloys can be used or isotope separation. Increasing the weight percentage of molybdenum in the alloys increases the amount of deuterium absorbed from the gas mixture, which then remains in the alloy in the form of a solid solution. Under the same conditions, little or no hydrogen was absorbed. Wang et al. [4] suppose that this selectivity is due to the fact that hydrogen and deuterium have a preference for different sites in the lattice of the alloys.

Zhao et al. [5] studied Ti–Mo alloy hydrides in the composition range 5 to 40 wt % molybdenum (4 to 30 at % Mo) and analyzed variations in their lattice parameters. The body-centered cubic (BCC) lattice parameter of unhydrided Ti–Mo alloys decreases with increasing Mo content. After hydriding five samples of Ti–Mo alloys containing 5, 10, 20, 30, and 40 wt % molybdenum (4, 7, 16, 23, and 30 at % Mo), the hydrogen-to-metal atomic ratio in the resultant hydride phases was H/M = 0.99. X-ray diffraction characterization showed that all the samples of the hydride phases had an FCC lattice. The lattice parameter of the hydride phases was found to increase with increasing Mo content, reaching a maximum at ≈10 wt % Mo. Further increasing the Mo content of the alloys reduces their lattice parameter. At the same time, the BCC lattice parameter of the unhydrided Ti–Mo alloys decreases with increasing Mo content. At room temperature, the equilibrium pressure of the hydriding products gradually increases with increasing molybdenum content.

Using X-ray diffraction and scanning electron microscopy, Wu et al. [6] investigated structural changes induced in hydrided Ti–Mo alloys containing ≤15 at % molybdenum by hydrogen desorption during annealing. Their results indicate that hydrogen desorption from the δ-hydride phase of a Ti–Mo alloy with an FCC lattice, similar to the δ-hydride phase with an FCC lattice in the Ti–H_2 system, is accompanied by the formation of a β-phase with a BCC lattice. As the desorption temperature is lowered, the disordered δ-phase (at H/M ~ 0.55–0.85) transforms into an ε-phase with a tetragonal lattice, corresponding to the ε-phase in the Ti–H_2 system. At the same time, the hydrogen concentration in the compounds in question is intermediate between the hydrogen concentrations in the γ-phase and ε-phase in the Ti–H_2

system, being closer to that in the γ-phase. With increasing molybdenum content, the stability region of the ε-phase increases.

Zheng et al. [7] measured hydrogen absorption–desorption isotherms of Ti−Mo alloys containing Zr, Sc, Y, and Al as dopants. According to their results, the dopants influence the equilibrium pressure and the slope and extent of the plateau in the isotherms obtained.

Lynch and Reilly [8] experimentally measured 40°C hydrogen desorption isotherms at pressures of 10 MPa or lower for Ti−Mo alloy hydrides containing 23, 33, 50, and 66 at % molybdenum. Their results demonstrate that increasing the molybdenum content of the alloys raises the pressure at which a hydride phase is formed. For example, no plateau was detected in the 40°C isotherm of the alloy with the composition $TiMo_{1.96}$ (which corresponds to $Ti_{0.34}Mo_{0.66}$), whereas the isotherms of the other compounds had a sloping plateau. The isotherms obtained for the systems studied were used to calculate their thermodynamic functions. The enthalpy of the β−γ phase transition in alloy−hydrogen systems was shown to decrease in the order Ti_2Mo ($Ti_{0.67}Mo_{0.33}$) > $Ti_{1.24}Mo$ ($Ti_{0.55}Mo_{0.45}$) > TiMo ($Ti_{0.50}Mo_{0.50}$), confirming that the stability of the hydride phases decreases with increasing Mo content. X-ray diffraction characterization of hydride phase samples showed that they had a slightly expanded BCC lattice in the case of the formation of the β-solid solution phase and an FCC lattice in the case of the γ-phase. Note that the lattice parameter of the β-phase was found to decrease with an increase in the molybdenum content of the starting alloy.

The structure of hydride samples resulting from hydrogen desorption from a δ-hydride phase with an FCC lattice based on Ti−Mo alloys was studied by Yuan et al. [9] using in situ X-ray diffraction. Hydrides were synthesized for a series of alloys containing 5, 10, 15, 20, 25, and 40 wt % molybdenum (4, 7, 11, 16, 20, and 30 at % Mo). The hydrogen desorption data obtained for the hydrides demonstrate that, in the temperature range 293–1173 K, the δ-hydride phase transforms into a β-hydride phase with a BCC lattice. Increasing the Mo content of the alloys reduces the lattice parameters of the δ- and β-hydride phases.

Xiao-Ming et al. [10] used small-angle X-ray scattering to probe the structure of samples of Ti−Mo alloy hydrides containing 5 to 20 wt % molybdenum (4 to 6 at % Mo). The samples of the hydride phases were found to have an FCC lattice, whereas all of the unhydrided alloys had a BCC lattice, except for the alloy containing 5 wt % Mo (4 at %), which had a hexagonal structure.

A number of groups studied the structure of hydride phases based on Ti−Mo alloys not only by X-ray diffraction but also by neutron diffraction because of the enhanced sensitivity of neutrons to hydrogen and, especially, to deuterium. For example, Wu et al. [11] investigated the structure of a deuterated Ti−Mo alloy containing 33 at % molybdenum. This composition of the alloy allowed them to obtain a neutron diffraction pattern of only the deuterium sublattice. Based on the results obtained, they identified a δ-phase with an FCC lattice in which the deuterium atoms occupied tetrahedral sites.

Sun et al. [12] used neutron diffraction to study the structure of deuterated Ti−Mo alloys containing 5 to 40 at % Mo. Their results demonstrate the formation of deuterides with D/M = 0.8−0.85 at a pressure of 15 MPa. The deuterides are isostructural with the δ-titanium hydride phase with an FCC lattice. The lattice parameter of the deuterides decreases with an increase in the molybdenum content of the alloys. Analysis of line broadening in neutron diffraction patterns showed that the distortion of the metal sublattice in the deuterides depended on the molybdenum content of the undeuterated alloys.

Based on published data, it is reasonable to assume that high hydrogen pressures may offer the possibility of synthesizing Ti−Mo alloy hydride phases with high Mo content. In this paper, we report a study of the $Ti_{0.40}Mo_{0.60}−H_2$ and $Ti_{0.34}Mo_{0.66}−H_2$ systems by X-ray and neutron diffraction at a high hydrogen pressure.

EXPERIMENTAL

Alloys for this investigation were prepared by reacting pure metals in an electric arc furnace under an inert atmosphere. Hydrides were synthesized and equilibria in alloy−hydrogen systems were investigated at hydrogen pressures of up to 250 MPa and temperatures of 20, −20, and −35°C. The experimental setup and measurement procedure were similar to those described previously [13]. X-ray diffraction measurements were made on Guinier and Rigaku diffractometers (CuK_α radiation). Before X-ray diffraction characterization, samples of the hydrides synthesized at high pressures were first passivated in air. To this end, the autoclave containing the sample was cooled to liquid-nitrogen temperature (77 K) under a high hydrogen pressure and then the pressure was released to atmospheric one. After that, the autoclave containing the sample was held open in air for 1 h in liquid nitrogen. As a result, the sample in the autoclave was in contact with air near liquid-nitrogen temperature (77 K).

The amount of hydrogen in the hydride samples was determined by high-temperature vacuum extraction. The sites occupied by hydrogen in the lattice of the hydrides were identified by neutron diffraction. To reduce incoherent scattering, deuterium was use instead of hydrogen and neutron diffraction data were obtained for deuterated samples. Our experiments were carried out on a high-resolution Fourier diffractometer (IBR-2 reactor, time-of-flight measurements) at the Joint Institute for Nuclear Research (Dubna).

Table 1. Lattice parameters of the $Ti_{0.34}Mo_{0.66}$ and $Ti_{0.40}Mo_{0.60}$ alloys and their hydriding products

Sample	Lattice	a, nm	$V \times 10^3$, nm^3	$\Delta V/V$, %
$Ti_{0.40}Mo_{0.60}$	BCC	0.3176(2)	32.0	–
$Ti_{0.40}Mo_{0.60}H_{1.1}$*	BCC	0.3257(2)	35.0	9
	FCC	0.4426(2)	87.0	36
$Ti_{0.34}Mo_{0.66}$	BCC	0.3166(3)	31.7	–
$Ti_{0.34}Mo_{0.66}H_{0.8}$*	BCC	0.3236(3)	33.9	7
	FCC	0.4378 (3)	83.9	32

* Hydrogen content was determined by high-temperature hydrogen extraction.

RESULTS AND DISCUSSION

It is known from data in the literature [14, 15] that, at high temperatures (above 1000°C), the Ti−Mo system contains a solid solution over the entire composition range. According to X-ray diffraction data, the $Ti_{0.40}Mo_{0.60}$ and $Ti_{0.34}Mo_{0.66}$ alloys prepared in this study had a BCC lattice with parameters similar to those reported by Lynch and Reilly [8]. Comparison of the lattice parameters of the alloys indicates that, with increasing molybdenum content, the lattice parameters decrease systematically (Table 1).

Figure 1 shows hydrogen absorption/desorption isotherms for the $Ti_{0.34}Mo_{0.66}$−H_2 and $Ti_{0.40}Mo_{0.60}$−H_2 systems. In contrast to metallic titanium, the $Ti_{0.34}Mo_{0.66}$ and $Ti_{0.40}Mo_{0.60}$ alloys react with hydrogen even in the first cycle at room temperature and pressures in the range 5−10 MPa at a high rate, essentially with no induction period. The isotherms for the $Ti_{0.40}Mo_{0.60}$−H_2 system at a temperature of 20°C contain two characteristic portions. At room temperature and pressures below 43 MPa, we observe hydrogen absorption and the formation of a hydride phase with the composition $Ti_{0.40}Mo_{0.60}H_{0.5}$. Further increase in pressure is accompanied by an additional hydrogen absorption, leading to the formation of a hydride phase with the composition $Ti_{0.40}Mo_{0.60}H_{1.0}$ at 210 MPa. At pressures in the range 1−80 MPa, the hydrogen absorption reaction is reversible; that is, the absorbed hydrogen is released as the pressure is reduced. At a reduced temperature (−35°C), the hydrogen content of the hydride phase increases to H/M = 1.1−1.2.

Increasing the molybdenum content of the alloy leads to significant changes in hydrogen desorption isotherms. As follows from the data presented in Fig. 1, the highest hydrogen content of the hydride phase in the $Ti_{0.34}Mo_{0.66}$−H_2 system at 180 MPa (−20°C) corresponds to the composition H/M = 0.5. As in the case of $Ti_{0.40}Mo_{0.60}$, hydrogen absorption in $Ti_{0.34}Mo_{0.66}$ is reversible in this range of pressures and temperatures. The above data lead us to assume that further lowering the temperature will increase the amount of hydrogen absorbed by the solid phase in the

$Ti_{0.34}Mo_{0.66}$−H_2 system. Lynch and Reilly [8] reported that the hydrogen content of an alloy with the same composition at a pressure of 10 MPa and temperature of 40°C corresponded to H/M = 1.0.

It follows from the present X-ray diffraction data (Table 1) that high hydrogen pressures in the $Ti_{0.40}Mo_{0.60}$−H_2 and $Ti_{0.34}Mo_{0.66}$−H_2 systems lead to the formation of a phase with an FCC lattice of the CaF_2 type. It is worth noting that, in this study, like in that reported by Lynch and Reilly [8], hydrogen absorption in $Ti_{0.34}Mo_{0.66}$ caused no transformation of the structure of the hydride phase into a Laves phase structure, similar to the hydride phase of the intermetallic compound $ZrMo_2$ studied by Semenenko et al. [14]. The present X-ray diffraction data made it possible to determine the phase composition of the synthesized hydride phases based on the $Ti_{0.34}Mo_{0.66}$ and
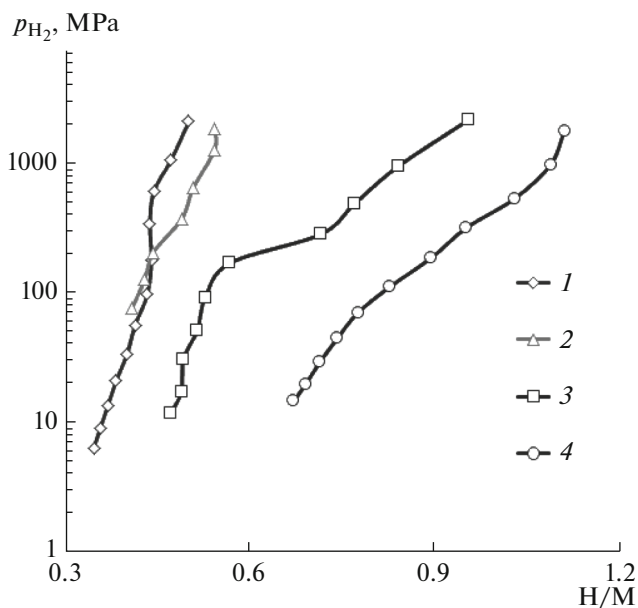
Fig. 1. Hydrogen desorption isotherms in the $Ti_{0.34}Mo_{0.66}$−H_2 system at temperatures of (*1*) 20 and (*2*) −20°C and in the $Ti_{0.40}Mo_{0.60}$−H_2 system at (*3*) 20 and (*4*) −35°C.
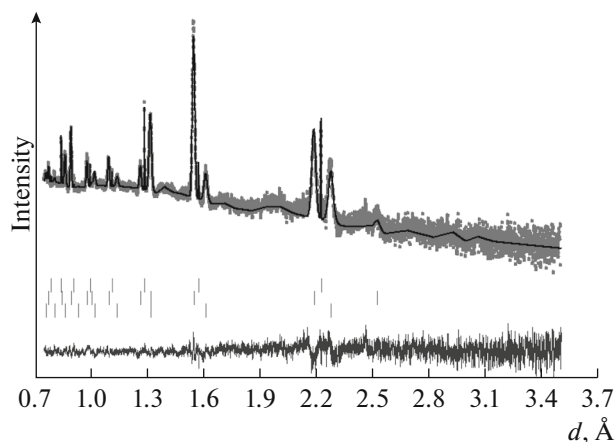
Fig. 2. Neutron diffraction pattern of the deuterated $Ti_{0.34}Mo_{0.66}$ alloy and Rietveld refinement profile ($R_w = 3.8\%$): raw data (points), calculated profile (upper continuous line), and difference plot (lower continuous line). The upper, medium, and lower vertical tick marks show the positions of the reflections from the phase with the BCC lattice, the phase with the FCC lattice, and the phase with the expanded BCC lattice, respectively.

$Ti_{0.40}Mo_{0.60}$ alloys. The fraction of the phase with an FCC lattice was determined to be about 50% in both samples. The hydrogen content of both samples was determined as well by high-temperature hydrogen extraction. The hydrogen content of the samples was found to be slightly lower than that extracted from experimental isotherms. The reason for this is that the hydride phases are unstable and even pyrophoric: they rapidly decompose in air at room temperature and the powder smoulders. Because of this, when powder samples are withdrawn from a reactor, the hydrides lose some of the hydrogen, in spite of the preliminary low-temperature passivation in air. The relative increase in the lattice volume, $\Delta V/V$, of the hydride phases with the CaF_2 structure exceeds 30% (Table 1).

The small increase in the volume of the BCC lattice (7−9%) in the samples of the hydride phases compared to the unhydrided alloys is most likely due to their low hydrogen content.

Neutron diffraction data were obtained for a deuteride sample based on the $Ti_{0.34}Mo_{0.66}$ alloy containing the largest amount of hydrogen, as shown by premeasured hydrogen absorption isotherms. Neutron diffraction results (neutron diffraction pattern in Fig. 2 and structural data in Tables 2 and 3) demonstrate that the deuterated sample consists of three phases. Two phases have a BCC lattice with parameters $a = 0.3170$ nm (14%) and $a = 0.3204$ nm (74%). The third phase has an FCC lattice of the CaF_2 type (12%) with a parameter $a = 0.4369$ nm. The deuterium atoms are located on interstitial sites of the FCC and BCC lattices, whereas the other BCC lattice (with a parameter $a = 0.3170$ nm) contains few or no deuterium atoms. In the FCC lattice, the deuterium atoms occupy tetrahedral sites surrounded by Ti(Mo) metal atoms. In the BCC lattice, the deuterium atoms also occupy tetrahedral sites. Calculations with the use of the neutron diffraction data showed that the deuteride phase with the FCC lattice had D/M = 1.00, whereas the phase with the BCC lattice had D/M = 0.15. Thus, the composition of the deuteride with the FCC lattice corresponds to $Ti_{0.34}Mo_{0.66}D_{1.0}$. According to the present neutron diffraction data, the relative increase in lattice volume, $\Delta V/V$, upon the formation of the FCC hydride phase is 26%. The presence of three phases in the deuteride, in contrast to the hydride samples, which consisted of just two phases according to X-ray diffraction data, is probably also due to the significant instability of the samples studied.

Comparison of the bond distances in the $Ti_{0.34}Mo_{0.66}D_{1.0}$ deuteride with data in the literature (for the $TiD_{1.98}$ deuteride [16] and $MoH_{0.95}$ hydride [17]) showed that the distances between the Ti(Mo) metal atoms decreased systematically because of the smaller atomic radius of molybdenum (0.140 nm) compared to titanium (0.149 nm). The Ti(Mo)−D

Table 2. Neutron diffraction data for the deuterated $Ti_{0.34}Mo_{0.66}$ alloy

Atom	Position	Occupancy	Atomic coordinates		
			x	y	z
BCC					
Ti	2a	0.3	0	0	0
Mo	2a	0.7	0	0	0
D	12d	0.3(3)	0.25	0	0.5
FCC					
Ti	2a	0.3	0	0	0
Mo	2a	0.7	0	0	0
D	8c	2.0(3)	0.25	0.25	0.25

$R_w = 3.8\%$.

Table 3. Bond distances in the $Ti_{0.34}Mo_{0.66}D_{0.1}$ deuteride

Compound	Atoms	Bond distances, nm
$Ti_{0.34}Mo_{0.66}D_{1.0}$	Mo−Ti(Mo)	0.3089(3)
	Ti(Mo)−D	0.1892(2)
	D−D	0.2185(3)
$Ti_{0.85}Mo_{0.15}D_{1.47}$ [18]	Mo−Ti(Mo)	0.3036
		0.3206
	Ti(Mo)−D	0.1889
	D−D	0.2027
		0.2020
$TiD_{1.98}$ [16]	Ti−Ti	0.3140
	Ti−D	0.1923
	D−D	0.2220
$MoH_{0.95}$ [17]	Mo−Mo	0.2379
	Mo−H	0.2071
	H−H	0.2921

bond distances are shorter than those in both the titanium deuteride and molybdenum hydride. The D−D bond distances are only slightly smaller than those in the titanium deuteride and molybdenum hydride (Table 3). The metal−metal and metal−deuterium bond distances in the $Ti_{0.34}Mo_{0.66}D_{1.0}$ deuteride studied here are intermediate between the largest and smallest distances in the $Ti_{0.85}Mo_{0.15}D_{1.47}$ deuteride, with a larger percentage of titanium, studied by Xu et al. [18]. The D−D bond distances in the lattice of the deuteride obtained in this study are longer than those in the $Ti_{0.85}Mo_{0.15}D_{1.47}$ deuteride.

Analysis of the calculated bond distances in the $Ti_{0.34}Mo_{0.66}D_{1.0}$ deuteride indicates that, like binary titanium and molybdenum hydrides, it retained metallic bonding between its atoms. The present neutron diffraction results agree with data reported by Sun et al. [12], who also pointed out a tendency for the metal−metal bond distances to decrease with increasing molybdenum content in a series of Ti−Mo alloy deuterides with a maximum molybdenum content of 40 at %. Note that a high molybdenum content of an alloy and, accordingly, a decrease in lattice parameter and in the associated size of the tetrahedral sites occupied by hydrogen lead to a decrease in the stability of the hydride phase, with an increase in equilibrium hydrogen pressure.

CONCLUSIONS

We have studied the hydriding of the $Ti_{0.40}Mo_{0.60}$ and $Ti_{0.34}Mo_{0.66}$ alloys at hydrogen pressures of up to 250 MPa. The hydrogen desorption isotherms obtained at temperatures of 20, −20, and −35°C indi-

cated the formation of $Ti_{0.40}Mo_{0.60}H_{1.1}$ and $Ti_{0.34}Mo_{0.66}H_{0.8}$ hydride phases.

X-ray diffraction data show that the hydride samples consist of phases with BCC and FCC (CaF_2 structure) lattices.

Neutron diffraction data for the $Ti_{0.34}Mo_{0.66}D_{1.0}$ deuteride sample at 77 K demonstrate that it contains a phase with the CaF_2 structure and two phases with a BCC lattice. The deuterium atoms are distributed over the FCC and one of the BCC lattices, whereas the other BCC lattice contains little or no deuterium.

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