

Article

Study of the Structural and Phase State and Hydrogen Interaction of a TiZrNbVTa High-Entropy Alloy Prepared by SPS

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Abstract

The effects of post-processing heat treatment on the structural and phase state and hydrogen sorption characteristics of an equiatomic TiZrNbVTa high-entropy alloy produced by mechanical activation and spark plasma sintering were investigated. The powder mixture was mechanically activated under argon and consolidated at 1450 °C. Two processing conditions were compared: SPS with a 7 min hold without subsequent treatment, and SPS with a 5 min hold followed by annealing at 1050 °C for 5 h. Phase composition was analyzed by X-ray diffraction. Hydrogenation was performed at 400–600 °C under a hydrogen pressure of 37–40 bar. Hydrogen content was evaluated using the SOAK kinetic method and inert gas fusion analysis with a RHEN-602 analyzer. Both materials were predominantly composed of a Nb-enriched body-centered cubic phase, whereas their secondary-phase compositions differed. After annealing, weak reflections tentatively attributed to ZrO₂ and TaO₂ appeared, whereas reflections previously assigned to Zr, V, and TiO₂ could no longer be resolved. According to the RHEN-602 measurements, the hydrogen content ranged from 1.12 to 1.37 wt.% for the SPS sample and from 0.64 to 2.08 wt.% for the SPS-HT sample between 400 and 600 °C. These results indicate that processing involving high-temperature annealing modifies the phase state and may enhance hydrogen uptake at 500–600 °C.

Keywords: high-entropy alloy; TiZrNbVTa; spark plasma sintering; SPS; heat treatment; X-ray diffraction; hydrogen sorption; thermal desorption spectroscopy



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1. Introduction

In recent decades, reducing dependence on fossil fuels has become particularly important because of increasing environmental pollution and worsening ecological problems [1–3]. To reduce the consumption of hydrocarbon resources, which are among the major sources of harmful emissions, the transition to hydrogen energy is being actively investigated [4,5]. The growing interest in hydrogen stems from its widespread availability and high specific energy density, that is, its ability to store a considerable amount

of chemical energy per unit mass. The development of hydrogen energy technologies requires efficient and safe materials for hydrogen storage [6]. One promising approach is solid-state hydrogen storage in metallic alloys capable of reversibly absorbing and releasing hydrogen through the formation of hydride phases [7–10]. Compared with the storage of gaseous hydrogen under high pressure, this approach offers improved safety and the potential to achieve a high volumetric hydrogen density [11]. High-entropy alloys are of particular interest in this field because their multicomponent composition enables the targeted modification of their crystal structure, phase stability, and hydrogen sorption characteristics [12,13].

High-entropy alloys are multicomponent systems in which the combination of several principal elements enables the formation of solid solutions characterized by pronounced lattice distortion and a wide variety of local atomic environments [14,15]. Alloys with a body-centered cubic (BCC) structure are of particular interest for hydrogen storage because of their relatively open interstitial configuration. Sahlberg et al. demonstrated the high hydrogen storage capacity of the multicomponent TiVZrNbHf alloy [16]. Subsequent studies confirmed that hydrogen sorption behavior is governed by local lattice distortion, phase composition, and chemical heterogeneity [17,18].

The Ti–Zr–Nb–V–Ta system combines elements with a high chemical affinity for hydrogen. The formation of hydride phases and a considerable hydrogen storage capacity were reported for the compositionally similar TiZrNbHfTa alloy [19]. For TiZrNbTa(Mo,V) systems, variations in the vanadium content were shown to affect the fraction of the cubic phase and the hydrogen content. Therefore, the properties of TiZrNbVTa may be controlled not only through its composition but also through its processing state.

The properties of high-entropy materials depend strongly on their processing route. The use of conventional melting methods for refractory components may result in chemical and structural heterogeneity within the material [20]. Spark plasma sintering (SPS) enables the consolidation of powder materials under the simultaneous effects of electric current, elevated temperature, and mechanical pressure. The high heating rate and short processing time promote rapid densification of the powder mixture, limit grain growth, and facilitate the production of dense samples with a controlled microstructure [21,22].

At the same time, the structural and phase state of SPS-processed materials depends on the sintering temperature and holding time, applied pressure, particle size of the initial powders, and degree of interdiffusion between the constituent elements [23,24]. The retention of residual phases, formation of solid solutions, and presence of secondary compounds may affect the interaction of the material with hydrogen [25,26]. Hydrogenation is accompanied by lattice expansion, phase transformations, hydride formation, and the development of internal stresses [27,28]. Therefore, establishing the relationship between the initial structure of the material and the changes occurring during hydrogen absorption is of considerable scientific and practical importance [29].

The aim of the present work is to compare the structural-phase state, hydrogen absorption, and thermal desorption behavior of equiatomic TiZrNbVTa in two technological states: after SPS and after SPS followed by high-temperature annealing.

Because the compared samples differed in both the SPS holding time and the presence of subsequent heat treatment, the observed difference in hydrogen content cannot be attributed exclusively to annealing. Differences in the degree of densification, residual porosity, chemical homogeneity, defect density, and impurity or oxide content may also have affected the measured results. Therefore, the findings should be interpreted as a comparison between two distinct processing states of the material rather than as a controlled assessment of the isolated effect of annealing.

2. Materials and Methods

2.1. Sample Preparation

An equiatomic Ti–Zr–Nb–V–Ta powder mixture was prepared from stoichiometric amounts of elemental Ti (99.995% purity, Chempur), V (99.95% purity, MRC), Zr (99.8% purity, Chempur), Nb (99.8% purity, Cerac), and Ta (99.6% purity, Johnson Matthey, Materials and Technology, UK) powders. The components were weighed using a Mettler Toledo microbalance with an accuracy of ± 0.0001 g.

Preliminary mixing was performed for 15 min at 200 rpm to ensure initial homogeneity. For mechanical activation, approximately 9 g of the resulting mixture was loaded into a 45 mL steel milling vial together with 10 mm diameter stainless-steel balls. The ball-to-powder mass ratio (BPR) was 10:1. All loading operations were performed in a VGB-3C glovebox under an argon atmosphere to prevent powder oxidation. After loading, the milling vial was hermetically sealed and installed in a Pulverisette 7 premium line planetary micro mill (Fritsch GmbH, Germany). Mechanical activation was conducted for 5 h at a rotational speed of 350 rpm under an argon atmosphere, with the addition of 2.5–3.0 wt.% stearic acid as a process control agent.

The powder mechanically activated for 5 h was used for consolidation. The powder was placed in a graphite die and sintered using a Dr. Sinter SPS-625 system at 1450 °C under a pressure of 60 MPa, with a heating rate of 100 °C/min, in a vacuum of 2–5 Pa. The temperature was monitored using a radiation pyrometer, while shrinkage and shrinkage rate were recorded using an integrated dilatometer. Two processing conditions were investigated (Table 1).

Table 1. Sample preparation conditions.

Sample	SPS Conditions	Subsequent Treatment	Designation
Nº1	1450 °C, 7 min	None	TiZrNbVTa-SPS
Nº2	1450 °C, 5 min	1050 °C, 5 h	TiZrNbVTa-SPS-HT

2.2. Research Methods

The phase composition was investigated using an XRD-7000S diffractometer (Shimadzu, Japan) in Bragg–Brentano geometry with CuK α radiation ($\lambda = 0.154$ nm), at a voltage of 40 kV and a current of 30 mA. The scanning range was $2\theta = 5$ – 90° . The phases were identified by comparing the experimental reflections with reference X-ray diffraction data. Hydrogenation was carried out using an automated Gas Reaction Automated Machine (GRAM) at 400, 500, and 600 °C. The hydrogen pressure was 37–40 bar, and the holding time was 12,000–15,000 s. The amount of absorbed hydrogen was recorded based on the gas-phase parameters (SOAK). Independent determination of the total hydrogen content was performed by inert gas fusion of the sample using an RHEN-602 analyzer (LECO Corporation, USA).

Thermally stimulated desorption of TiZrNbVTa-SPS was investigated under a vacuum of 10^{-9} – 10^{-7} Torr with heating to 475 °C at a rate of 6 °C/min. The H₂ release intensity was recorded using an RGA ExTorr XT300 mass spectrometer. The sample mass was 0.1602 g.

3. Results and Discussion

3.1. Characterization of the Morphology and Structural-Phase State of the Materials

To determine the phase composition and assess the structural state of the TiZrNbVTa alloy of the first series, X-ray phase analysis was performed. The obtained results made it possible to identify the main crystalline phases present in the investigated material. The diffraction patterns of the investigated samples are presented in Figure 1.

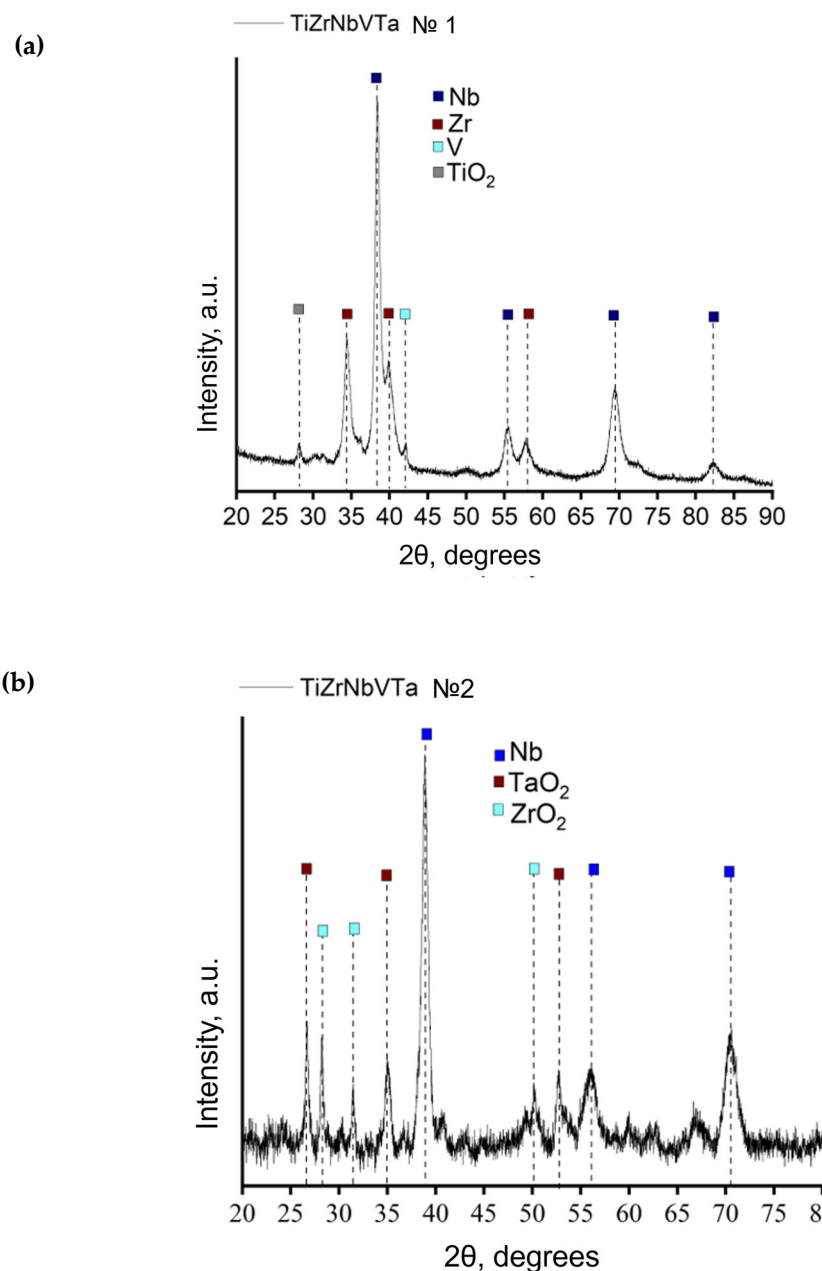


Figure 1. X-ray diffraction patterns of the TiZrNbVTa samples: (a) sample No. 1 produced by SPS at 1450 °C for 7 min without subsequent heat treatment; (b) sample No. 2 produced by SPS at 1450 °C for 5 min and subsequently annealed at 1050 °C for 5 h.

According to the X-ray diffraction analysis, the samples have the same elemental composition but differ in their phase composition. In both samples, the major phase was identified and labeled as Nb in the diffraction patterns. Secondary phases were also detected, and their assemblage depended on the material processing conditions. The diffraction pattern of Sample No. 1 (Figure 1a) contains reflections attributed to Zr, V, and TiO_2 phases. After annealing, these reflections could no longer be resolved, whereas weak reflections tentatively assigned to ZrO_2 and TaO_2 appeared. Owing to their low intensity and possible peak overlap, these phase assignments remain uncertain. The observed differences may also be associated with surface oxidation, the presence of impurities, or a decrease in the content of individual phases below the detection limit of X-ray diffraction. Therefore, the obtained data indicate a possible change in the phase state after heat treatment but do not confirm the formation or complete disappearance of

specific secondary phases. Quantitative full-pattern analysis and additional structural and chemical investigations are required to verify these phase transformations.

3.2. Evaluation of the Interaction with Hydrogen

The interaction of the materials with hydrogen was investigated at temperatures of 400, 500, and 600 °C. The hydrogen pressure in the reaction chamber was maintained at 40 bar. The duration of the experiments ranged from 12,000 to 15,000 s. The kinetic curves of hydrogen absorption by the TiZrNbVTa samples are presented in Figure 2.

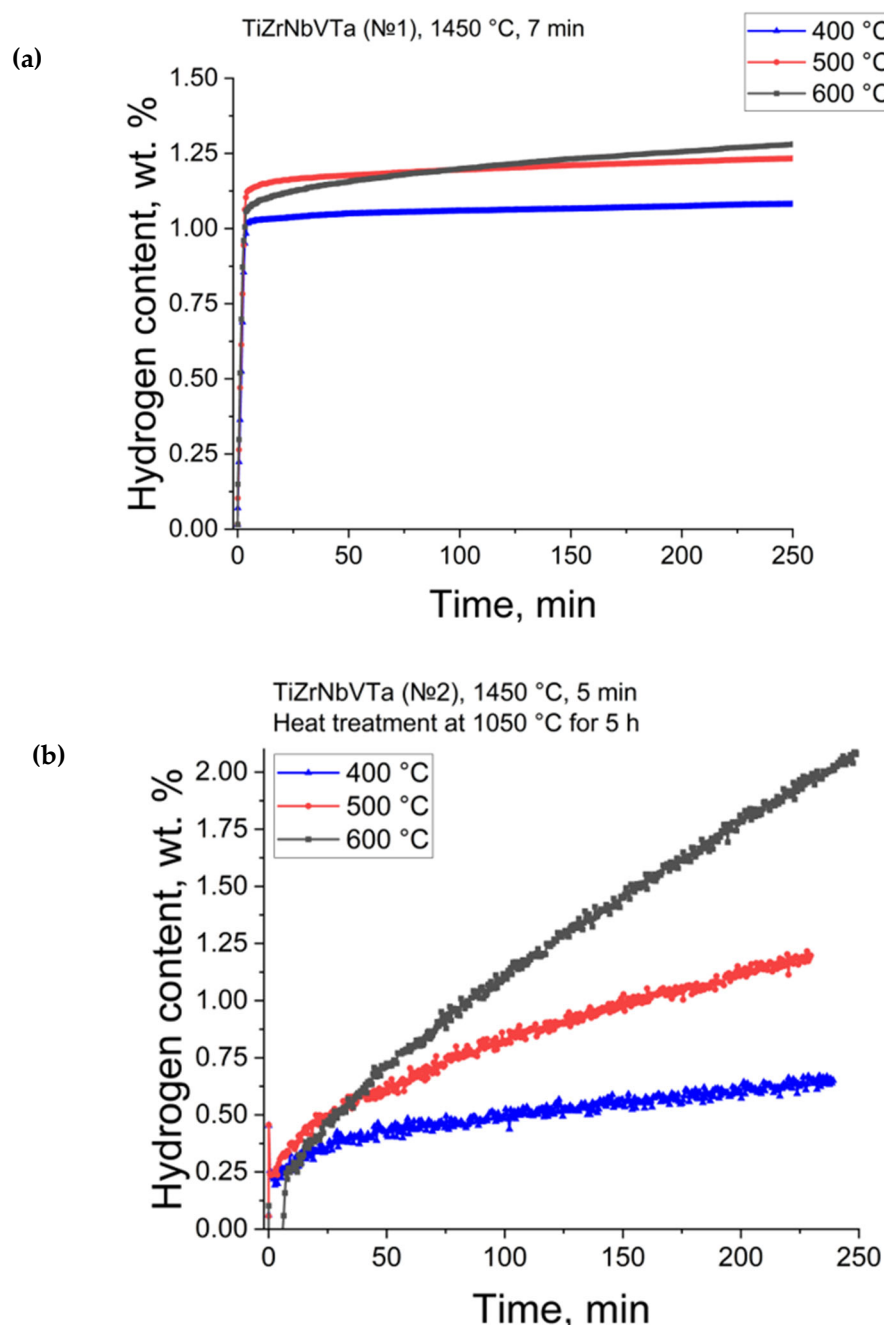


Figure 2. Kinetic hydrogen sorption curves of TiZrNbVTa: (a) at 1450 °C and 7 min; (b) at 1450 °C for 5 min followed by heat treatment at 1050 °C for 5 h.

Figure 2a presents the kinetic hydrogen absorption curves of the TiZrNbVTa sample obtained by SPS at 1450 °C with a holding time of 7 min without subsequent heat treatment. A sharp increase in the absorbed hydrogen content during the first minutes of the

experiment is characteristic of all investigated temperatures. Subsequently, the absorption rate decreases, and the curves gradually approach nearly steady-state values. The most pronounced increase in hydrogen content is observed with increasing temperature to 600 °C. During the initial interaction period, approximately within the first 25–30 min, the curve at 500 °C is located above the curve at 600 °C. This indicates a higher hydrogen absorption rate at 500 °C at this stage of the experiment. However, subsequently, the hydrogen content at 600 °C continues to increase and, by the end of the measurement, exceeds the values obtained at 400 and 500 °C. According to the data presented in Table 2, the hydrogen content determined by the inert gas fusion method using the RHEN-602 analyzer was:

- 1.12 wt.% at 400 °C;
- 1.23 wt.% at 500 °C;
- 1.37 wt.% at 600 °C.

Table 2. Characteristics of the Investigated Materials.

Nº	Composition	Temperature	RHEN-602 (Actual), wt. %	SOAK (37–40 bar, GRC1), wt. %	RHEN- 602/SOAK Ratio
1	TiZrNbVTa, SPS at 1450 °C for 5 min, followed by heat treatment at 1050 °C for 5 h; 1.1154 g (No. 2)	400 °C	0.64	0.4	1.6
		500 °C	1.19	0.74	1.6
		600 °C	2.08	1.30	1.6
2	TiZrNbVTa, SPS at 1450 °C for 7 min, without subsequent heat treatment; 6.3866 g (No. 1)	400 °C	1.12	0.68	1.6
		500 °C	1.23	0.75	1.6
		600 °C	1.37	0.85	1.6

Thus, for this sample, an increase in the hydrogenation temperature from 400 to 600 °C was accompanied by an increase in the hydrogen content determined by the RHEN-602 method. Figure 2b presents the kinetic hydrogen absorption curves of the TiZrNbVTa sample sintered at 1450 °C with a holding time of 5 min and subsequently annealed at 1050 °C for 5 h. Compared with the sample without subsequent heat treatment, the annealed material exhibits an increase in the absorbed hydrogen content at temperatures of 500 and 600 °C. The highest hydrogen content determined by the RHEN-602 method was 2.08 wt.% at 600 °C.

At 600 °C, the hydrogen content in the annealed sample is approximately 1.5 times higher than the corresponding value for the sample without subsequent heat treatment. At 500 °C, the decrease is approximately 0.96 times. At 400 °C, in contrast, a decrease in the hydrogen content from 1.12 to 0.64 wt.% is observed, corresponding to approximately 42.8%. The obtained results show that subsequent heat treatment has a different effect on the interaction of the TiZrNbVTa alloy with hydrogen over the investigated temperature range. The most pronounced increase in the absorbed hydrogen content after annealing is observed at 500 and 600 °C, whereas at 400 °C the hydrogen content decreases. The main characteristics of the investigated materials are presented in Table 2. The duration of the interaction of the samples with hydrogen was 12,000–15,000 s. At 600 °C, increased thermal activation of hydrogen dissociation at the surface, transport into the near-surface region, and subsequent bulk diffusion may partially overcome kinetic barriers, including those associated with surface oxides, thereby allowing hydrogen to access a larger fraction of the available sorption sites [30–32]. However, the available data do not allow the opposite behavior observed at 400 and 600 °C to be explained conclusively. Further systematic studies are required to clarify the underlying mechanisms.

Reversible hydride reactions at elevated temperatures may be relevant to stationary thermochemical energy-storage systems coupled to concentrated solar power plants or sources of high-grade industrial waste heat [33–35]. Metal hydrides operating at temperatures approaching or exceeding 600 °C have been proposed for next-generation solar-thermal plants, and operational high-temperature metal-hydride prototypes have already been demonstrated [35].

3.3. Thermal Desorption Behavior of the Hydrogenated Samples

The thermal desorption curve of TiZrNbVTa is characterized by a more complex shape and the presence of several features in different temperature ranges. In the low-temperature region, maxima and local changes in intensity are observed at temperatures of approximately 55, 68, 100, and 129 °C. The pronounced structure of the low-temperature peaks indicates the presence of several different types of hydrogen traps with similar binding energies. The TiZrNbVTa alloy (№ 1) is characterized by a wide range of hydrogen energy states from weakly bound surface states (55–129 °C) to more firmly retained bulk states (284–350 °C). These features indicate the heterogeneous nature of the hydrogen release process in this temperature range.

With further heating, the desorption intensity changes non-monotonically. In the region around 190 °C, an additional feature of the curve is observed, after which the intensity gradually increases. In the range of approximately 250–300 °C, a pronounced maximum is formed at a temperature of about 284 °C. After that, the desorption intensity decreases somewhat and then increases again. The most pronounced maximum is observed at a temperature of approximately 350 °C. After reaching this maximum, the hydrogen release intensity gradually decreases and, with further heating, approaches the initial level. Thus, the thermal desorption curve of TiZrNbVTa is characterized by the presence of several maxima in the temperature range from 55 to 350 °C. Figure 3b shows the thermodesorption spectroscopy profile for the TiZrNbVTa sample (№ 2), which is divided into two temperature regions. The low-temperature hydrogen release region (63–205 °C) is associated with the release of weakly bound hydrogen localized on the surface and in defects. The most pronounced peaks are at 100 and 164 °C. In the high-temperature region (300–481 °C), the main hydride phases decompose, which is associated with the release of hydrogen from the material's bulk (traps, defects). As with the first sample, the material is characterized by high thermal stability of hydrogen retention.

The present study was conducted within a temperature range of 400–600 °C and at hydrogen pressures of approximately 37–40 bar. These experimental conditions made it possible to identify the main trends in the hydrogen absorption behavior of the investigated TiZrNbVTa alloy. Nevertheless, further measurements over broader temperature and pressure ranges would provide a more comprehensive understanding of its hydrogen-storage performance. The present work compares two processing states of the alloy, demonstrating that the preparation conditions and subsequent heat treatment influence its structural and hydrogen absorption characteristics. Further systematic studies using samples produced under identical SPS conditions would help to clarify the specific contribution of annealing and other microstructural factors. The differences between the hydrogen contents determined by the SOAK and RHEN-602 methods may be related to the distinct measurement principles and experimental conditions employed. Future work will therefore focus on additional verification of the hydrogen capacity, absorption–desorption cycling, quantitative analysis of the thermal desorption spectra, determination of the reversible hydrogen capacity, and quantitative phase characterization before and after hydrogenation. These studies will complement the present results and support a more detailed assessment of the practical hydrogen-storage potential of the TiZrNbVTa alloy.

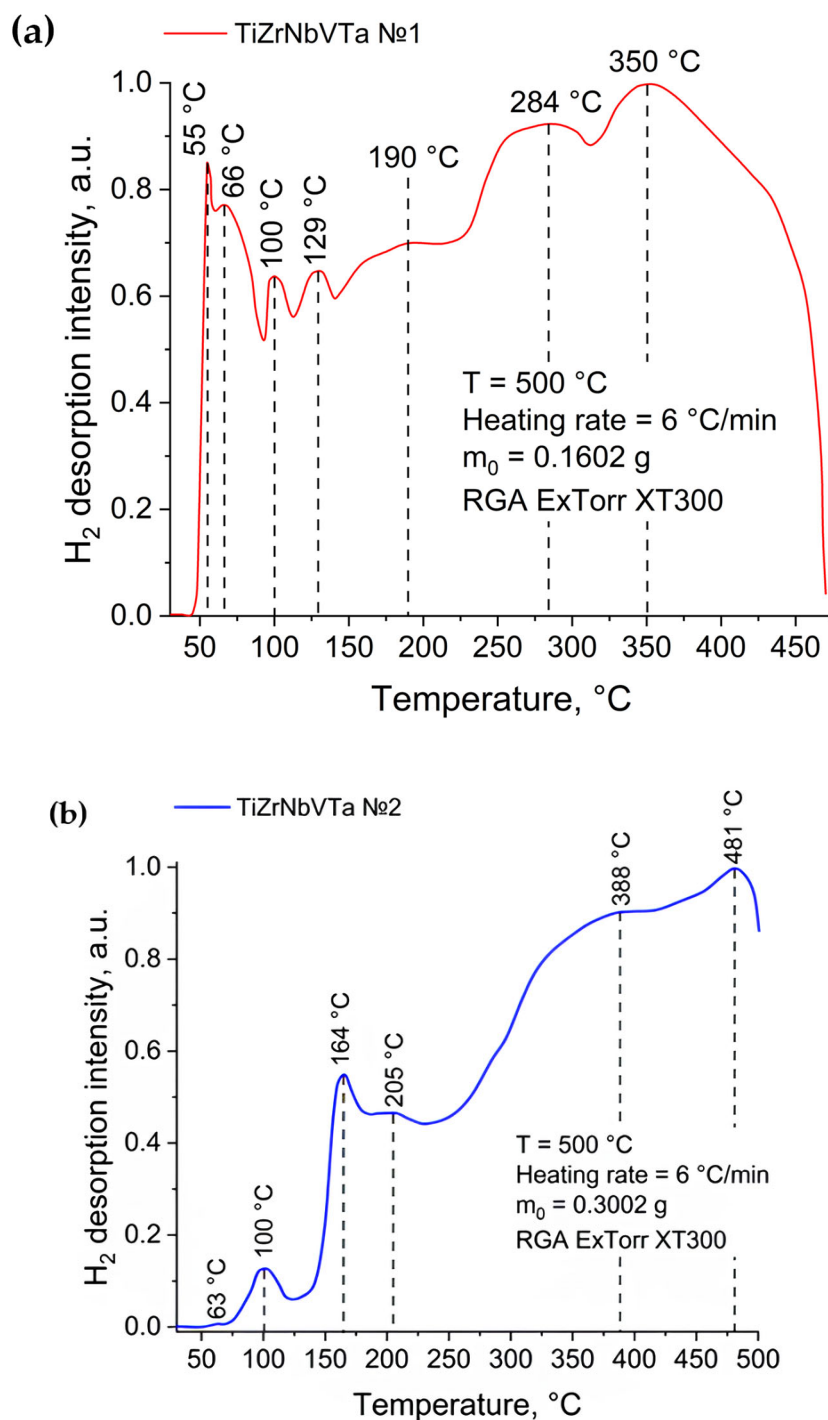


Figure 3. TDS curves for the TiZrNbVTa №1 (a) and №2 (b) samples.

4. Conclusions

The equiatomic TiZrNbVTa alloy after SPS and after SPS followed by annealing is characterized by the predominance of reflections of a Nb-like BCC phase and a different set of weak reflections of secondary phases. After annealing at 1050 °C for 5 h, reflections previously attributed to Zr, V, and TiO₂ could no longer be resolved, whereas weak reflections tentatively assigned to ZrO₂ and TaO₂ appeared. These assignments require confirmation by quantitative full-profile analysis.

According to the RHEN-602 measurements, the hydrogen content ranged from 1.12 to 1.37 wt.% in the SPS sample and from 0.64 to 2.08 wt.% in the SPS-HT sample between 400 and 600 °C. At 600 °C, the value for the SPS-HT sample was approximately 51.8% higher

than that for the SPS sample. However, the absence of a clearly defined plateau in the kinetic curves and the discrepancy between the RHEN-602 and SOAK results do not allow these values to be interpreted as confirmed equilibrium or reversible hydrogen-storage capacities.

The thermal desorption spectra of both processing states exhibit several hydrogen-release features, indicating energetically heterogeneous hydrogen binding. The SPS sample shows several maxima between 55 and 350 °C, whereas the SPS-HT sample exhibits low-temperature features between 63 and 205 °C and a broad high-temperature desorption region extending to approximately 481 °C. Overall, the results demonstrate the sensitivity of the structural and hydrogen-interaction characteristics of TiZrNbVTa to its processing state.

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Conflicts of Interest: The authors declare no conflicts of interest.

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