Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science. 2021 vol. 23 no. 3 pp. 100–111
ISSN: 1994-6309 (print) / 2541-819X (online)
DOI: 10.17212/1994-6309-2021-23.3-100-111

NSTU
NETI
Obrabotka metallov -
Metal Working and Material Science
Journal homepage: http://journals.nstu.ru/obrabotka_metallov

Hydrogen and its effect on the grinding of Ti-Ni powder

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ARTICLE INFO

Article history:
Received: 07 April 2021
Revised: 23 April 2021
Accepted: 30 July 2021
Available online: 15 September 2021

Keywords:
Ni-Ti powder
Mechanical treatment
Hydrogenation
Hydrogen
Coherently diffracting domain
Lattice parameter
Phase composition

Funding
The results were obtained in the framework of the Integrated Project “Establishment of import-substituting high-tech full-cycle production of complex-shaped indexable carbide cutting inserts for priority industries” (Agreement No. 075-11-2019-036 dated November 27, 2019) implemented by the ISPMS SB RAS at the financial support of the Ministry of Education and Science of the Russian Federation as part of Decree of the Government of the Russian Federation No. 218 dated April 09, 2010.

Acknowledgements
Research were conducted at core facility “Structure, mechanical and physical properties of materials”.

ABSTRACT

Introduction. Industrial nickel-titanium alloy PN55T45 closed to the equiatomic composition is widely used for the manufacture of products by powder metallurgy. To achieve high physical and mechanical properties of the material obtained by this method, it is necessary to use fine powders, which can be obtained by implementing high-intensity grinding in a planetary ball mill. However, during such treatment, contamination, powder oxidation and particle aggregation, etc. are possible. To solve this problem, preliminary hydrogenation is proposed for subsequent grinding in a planetary ball mill. **The aim of the work** is to study the effect of hydrogen on the grinding of titanium nickelide powder. **Materials and methods.** The morphology and average particle size of the powders were studied by scanning electron microscopy. The structure and phase composition of the powders were investigated by the methods of X-ray structural and X-ray phase analysis. The data of X-ray structural analysis were used to estimate the dislocation density. **Results and discussions**. It is shown that the use of pre-hydrogenation for 180 minutes before machining allows reducing the average particle size by about a half. After mechanical treatment of the powder, the parameters of the crystal lattices of the TiNi (austenite), Ti_2Ni and Ni_3Ti phases do not change within the error range. After mechanical treatment of the powder with preliminary hydrogenation, the crystal lattice parameter of only the Ti_2Ni phase changes significantly, in particular, at 180 minutes of hydrogenation, the lattice parameter increases to $1.1457 \pm 5\times10^{-4}$ nm, which corresponds to the stoichiometry of the $Ti_2NiH_{0.5}$ hydride with a lattice parameter of 1.1500 nm. The highest dislocation density estimated by X-ray diffraction analysis is contained in the Ti_2Ni (511) phase than in the TiNi (austenite) (110) and Ni_3Ti (202) phases. Thus, preliminary hydrogenation can be an effective method of powder grinding due to the formation of brittle hydride and suppression of the aggregation of fine particles during high-intensity mechanical treatment.

For citation: Abdulmenova E.V., Kulkov S.N. Hydrogen and its effect on the grinding of Ti-Ni powder. *Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science*, 2021, vol. 23, no. 3, pp. 100–111. DOI: 10.17212/1994-6309-2021-23.3-100-111. (In Russian).

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Introduction

It is known that the presence of hydrogen in a material has an ambiguous effect on its properties. In [1, 2], it is shown that hydrogen can reduce metal deformability, i.e. it makes it brittle [3] due to formation of brittle hydrides on the main slip and twin planes [4]. On the other hand, in [5], titanium alloy plasticity is found to increase due to the hydrogen plasticization effect, and in [6], it is shown that the presence of hydrogen in the lattice changes phase transformation temperatures.

It is known that plastic materials are able to deform without breaking the continuity and crack formation [7]. One of these materials is titanium nickelide, which possesses a number of unique properties [8], and is widely applied in industry. Ti-Ni products are often produced by powder metallurgy techniques. This technology employs fine powders to produce materials with high physical and mechanical properties [9]. Fine powders can be prepared by different methods, such as physicochemical (reduction method, electrolytic method, carbonyl method) and mechanical (production of powder from metal melt, solid state grinding by ball, vortex, planetary ball mills and vibrating mills) production methods. One of grinding methods is high-intensity mechanical treatment of powder in a planetary ball mill. This method is relatively inexpensive, fast and easy-to-implement; however, milling can involve contamination, powder oxidation and particle aggregation, etc. [10]. It is known that metal hydrides are typically brittle substances [11, 12], which can prevent aggregation of fine particles during high-intensity grinding. Therefore, pre-hydrogenation can not only intensify powder grinding, but powder hydrogenation-dehydrogenation can also cause a self-grinding effect due to lattice expansion–contraction [13]. Moreover, sintering of hydrogenated powders according to [14-16] leads to hydrogen release, which helps remove impurities such as oxygen, carbon, and chlorine, whereas, after milling, impurities remain in heated powders. This is confirmed in [17, 18], which report a positive effect of hydrogen during sintering followed by hydrogen released.

The **aim** of this study is to investigate the effect of hydrogen on Ti-Ni powder grinding.

The main **objectives** of this study are as follows:

1) to investigate the morphology of Ti-Ni particles and its size by scanning electron microscopy before and after mechanical treatment of the initial and hydrogenated powder; 2) to study the change in phase composition and parameters of the fine crystal structure by X-ray structural and X-ray phase analyses after mechanical treatment of the initial and hydrogenated powder.

Materials and methods

The industrial powder of titanium nickelide of the PN55T45 grade produced by the Polema company was studied. The powder was milled in an AGO-2 planetary ball mill (Russia) in air at room temperature and atmospheric pressure. The steel ball diameter was 0.7 cm, the ball-to-powder ratio was 1:5, and the rotation speed of the planetary disk was 1.820 rpm, which provides 60g acceleration. The treatment time was 100 seconds.

Hydrogenation before mechanical treatment was performed in a special cell by electrochemical method. The electrolyte was 20 % aqueous solution of sodium chloride (NaCl) and surfactant, dextrin $(C_6H_{10}O_5)_n$, at a concentration of 1.5 g/L at room temperature [19]. The cathode current density was 55 mA/cm^2, and the cell voltage was 4.0 V. A graphite plate was used as an anode, and powder, placed on a stainless-steel cup, was a cathode [20]. The hydrogenation time was 90 and 180 minutes.

The morphology and average powder particle size were estimated from SEM images obtained with a scanning electron microscope (SEM) using a TESCAN VEGA3 SBH microscope. The particle shape was estimated by the method proposed by Hausner [21]. Two dimensions of the projection plane of each particle (maximum d_{max} and minimum d_{min}) were measured to plot the particle size distributions; in total, about 650 particles were analyzed. The measurement data were used to calculate the average particle size, size dispersion and median diameter. The Ti-Ni powder in the initial state consists mainly of spherical particles with a smooth surface with an average size of 11.1 µm, and the size dispersion is 7.5 µm. The powder contains fractions of small and large particles. Most of the particles of the fine fraction are up to 7.3 µm in size, its amount is 37.2 % of the total, and agglomerated particles are up to 55 µm in size [22].

X-ray analysis was used to determine the structure and phase composition of the samples. XRD patterns were obtained using a DRON-type diffractometer (Russia) with CuK_α radiation with an exposure at each recording point to provide statistical accuracy of not less than 0.5 %. The ICDD PDF-2 powder database was used to identify phase composition. Crystal lattice parameters were determined using the rtp32 program for XRD calculations [23] from interplanar distances for all reflections in the angular range of 10°–100°. The relative phase content was determined from integral intensities of all identified phases in the angle range of 35°–50°; the most intense reflections of all phases are located in this range. The sum of all lines is 100 %. The size of the coherently diffracting domain / coherent scattering region (CDD/CSR) was calculated from the first line of X-ray spectra using the Scherrer equation [24]; for calculation, FWHM was determined for each phase. For powder, all reflections characteristic of the phases are difficult to determine due to significant broadening of X-ray diffraction lines; therefore, the Williamson-Hall method cannot be used. Nevertheless, it should be noted that the calculated CDD/CSR sizes are slightly underestimated since the contributions of the CDD/CSR size and microdistortion to broadening are not separated in this case. The diffraction profiles were approximated using the Lorentz function.

Results and discussion

Fig. 1 shows SEM images of powders and particle size distribution after mechanical treatment (a, b) and after mechanical treatment of the powder hydrogenated for 90 minutes (c, d), and 180 minutes (e, f). As can be seen, after mechanical treatment (Fig. 1, a), the average particle size decreased by 2 % and is 10.9 µm, while the size dispersion increased from 7.5 to 10.9 µm. In the powder, fractions of small and large particles can be distinguished (Fig. 1,b). The size of fine particles with a smooth surface is up to 3.6 µm, its number is 33.3 % of the total number, and agglomerated particles are up to 83.1 µm in size. After mechanical treatment with preliminary hydrogenation of the powder for 90 minutes (Fig. 1, c), the average particle size decreased by 13 % and is 9.7 µm, the size dispersion is 9.6 µm. Fine particles with a smooth surface are up to 3.6 µm in size, and its number increased to 37.6 % of the total number; agglomerated particles are up to 80.6 µm in size (Fig. 1,d). After mechanical treatment with preliminary hydrogenation of the powder for 180 minutes (Fig. 1, e), the average particle size decreased by 40 % and is 6.7 µm, the size dispersion is 7.7 µm. Fine particles with a smooth surface are up to 2.6 µm in size, its number is 41.2 % of the total, and the size of agglomerated particles decreased to 62.9 µm (Fig. 1,f). During mechanical treatment and pre-hydrogenation, the particle shape does not change and is close to spherical apparently due to the effect of high-energy grinding in the planetary mill.

Fig. 2 shows XRD patterns of the powders in the initial state, after mechanical treatment (sample No. 1), after mechanical treatment with pre-hydrogenation for 90 minutes (sample No. 2) and 180 minutes (sample No. 3). As can be seen, all XRD diffraction patterns contain diffraction reflections of the TiNi austenite phase, traces of the TiNi, Ti_2Ni, and Ni_3Ti martensitic phase. The XRD diffraction pattern obtained after mechanical treatment (MT) shows no change in phase composition (sample No. 1), while FWHM increased insignificantly, not more than by 19 %. The XRD diffraction patterns obtained for powders after MT with pre-hydrogenation (samples 2 and 3) show two intense peaks at angles 32.1° and 45.8°, which belong to the cubic phase of Ti_2NiH_x hydride with reflection indices (400) and (531).

The relative content of the Ti_2Ni phase increases from 36±5 % (powder in the initial state) to 42±5 %, and that of TiNi (austenite + martensite) decreases from 62±5 % (powder in the initial state) to 54±5 % during mechanical treatment of the powder hydrogenated for 180 minutes. After mechanical treatment of the initial powder and the powder hydrogenated for 90 minutes, the relative content of the latter virtually does not change with respect to the initial powder, all powders contain a small amount of the Ni_3Ti phase, not more than 5 %.

After hydrogenation, the angular positions of the lines of different phases are shifted (Fig. 2); therefore, the parameters of the crystal lattices of the phases are calculated. The calculation results are

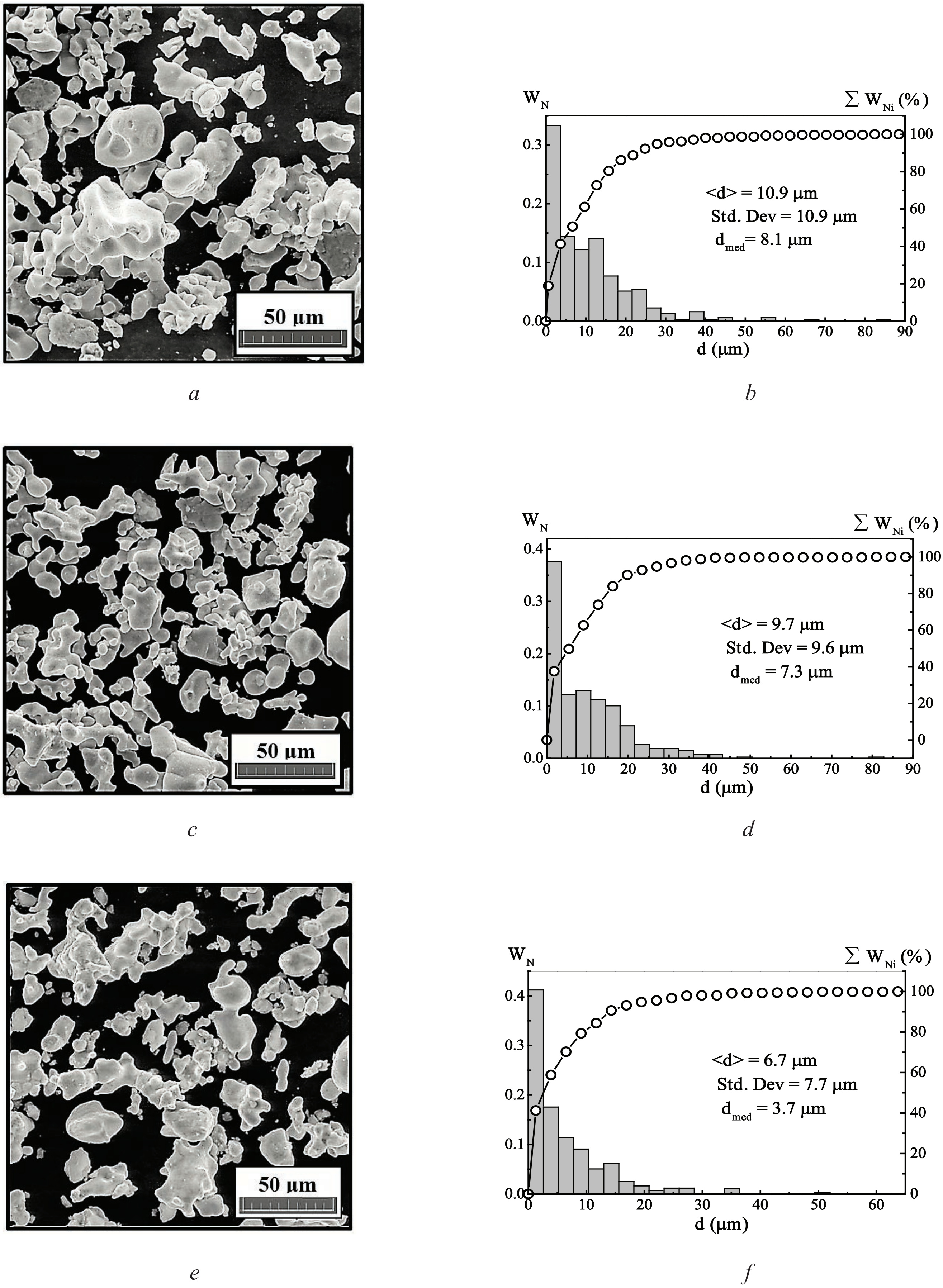

Fig. 1. SEM images and particle size distribution of the Ti-Ni powder after ball milling (*a*, *b*) and after mechanical treatment with pre-hydrogenation for 90 minutes (*c*, *d*), 180 minutes (*e*, *f*)

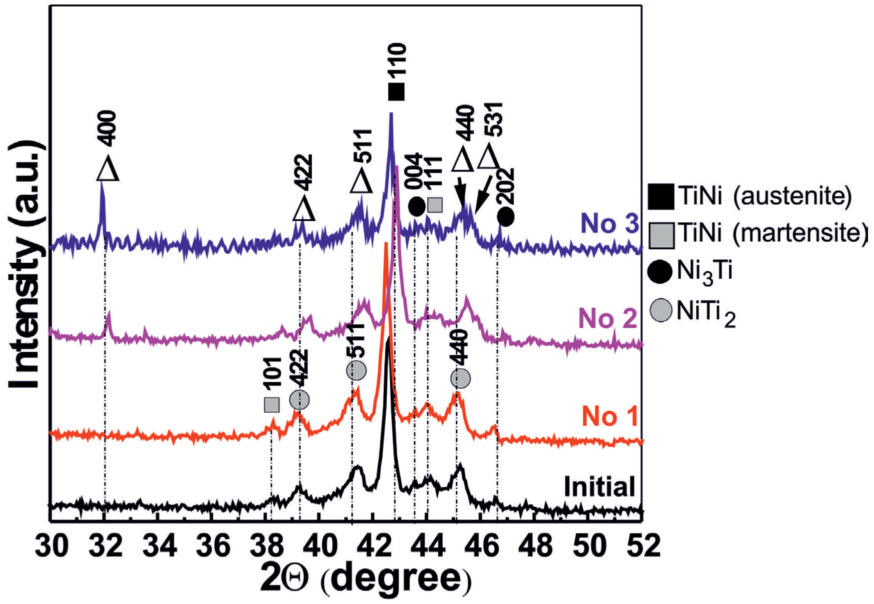

Fig. 2. XRD patterns of powders: in the initial state, sample No. 1 – after mechanical treatment, samples No. 2,3 – after mechanical treatment with pre-hydrogenation for 90 and 180 minutes, respectively

summarized in Table 1. After mechanical treatment of the initial powder and hydrogenated powder, the lattice parameter of the martensite phase is difficult to determine due to significant broadening of the X-ray diffraction lines; therefore, this value is indicated by a "question mark" in the table. As can be seen from the table, the parameters of the crystal lattices of all phases detected in the powder in the initial state are in good agreement with the literature data [25–28]. After mechanical treatment, the lattice parameters of TiNi (austenite), Ti_2Ni, and Ni_3Ti phases do not change within the error limits. After MT of the powder hydrogenated for 180 minutes, the lattice parameter of the Ni_3Ti phase decreases from 0.8316 nm (powder in the initial state) to 0.8241 nm, and the lattice parameter of the Ti_2Ni phase increases from 1.1333 nm (powder in the initial state) to 1.1457 nm. This means that during mechanical treatment of the hydrogenated powder, the lattice parameter of only Ti_2Ni phase changes significantly, and its value is close to the lattice parameter of hydride with $Ti_2NiH_{0.5}$ stoichiometry, which equals 1.1500 nm (ICDD PDF2 270346) [29, 30].

Table 2 shows data on microstructural parameters of phases. It also presents an estimate of the dislocation density (ρ) calculated in accordance with [31] from the first line of the X-ray spectra under the assumption that its width depends on the CDD/CSR size only; therefore, the density is overestimated. As can be seen from the table, FWHM of the TiNi (austenite) phase does not change within the error during mechanical treatment of the initial and hydrogenated powder. Whereas, FWHM of the Ni_3Ti phase increases from 0.084° (powder in the initial state) to 0.260° (powder hydrogenated for 90 minutes) and then decreases to 0.143° (powder hydrogenated for 180 minutes).

The CDD/CSR size calculated for the TiNi and Ti_2Ni phases does not change within the error range and is 29±5 nm and 11±5 nm for all powders, while for the Ni_3Ti phase after MT it decreases from 108±5 nm to 49±5 nm, after MT of the hydrogenated powders it decreases to 35±5 nm at 90 minutes, increases to 63±5 nm (180 minutes).

An absolute increase in the defect density in the Ni_3Ti phase exceeds that in TiNi and Ti_2Ni phases, however, the value of the dislocation density for the Ti_2Ni phase is 6 fold greater than that for the TiNi phase and 67 fold greater than that for the Ni_3Ti phase in the initial state. Apparently, high defect density in the Ti_2Ni phase in the initial state intensifies interaction of hydrogen with this phase [20], which also intensifies crushing during mechanical treatment due to formation of brittle hydride.

Table 1

Lattice parameters of the formed phases

Treatment type	Phase lattice parameter, nm
Initial state	TiNi (austenite): $a = 0.3007 \pm 5 \cdot 10^{-4}$
	TiNi (martensite): $a = 0.2690 \pm 5 \cdot 10^{-4}$ $b = 0.4570 \pm 5 \cdot 10^{-4}$ $c = 0.4122 \pm 5 \cdot 10^{-4}$ $\beta = 87.4°$
	Ti_2Ni: $a = 1.1333 \pm 5 \cdot 10^{-4}$
	Ni_3Ti: $a = 0.5099 \pm 5 \cdot 10^{-4}$ $c = 0.8308 \pm 5 \cdot 10^{-4}$ $c/a = 1.6293$
Mechanical treatment	TiNi (austenite): $a = 0.3009 \pm 5 \cdot 10^{-4}$
	TiNi (martensite): ?
	Ti_2Ni: $a = 1.1338 \pm 5 \cdot 10^{-4}$
	Ni_3Ti: $a = 0.5108 \pm 5 \cdot 10^{-4}$ $c = 0.8316 \pm 5 \cdot 10^{-4}$ $c/a = 1.6280$
Mechanical treatment with pre-hydrogenation for 90 minutes	TiNi (austenite): $a = 0{,}2995 \pm 5 \cdot 10^{-4}$
	TiNi (martensite): ?
	Ti_2Ni: $a = 1.1393 \pm 5{\cdot}10^{-4}$
	Ni_3Ti: $a = 0.5067 \pm 5 \cdot 10^{-4}$ $c = 0.8241 \pm 5 \cdot 10^{-4}$ $c/a = 1.6265$
Mechanical treatment with pre-hydrogenation for 180 minutes	TiNi (austenite): $a = 0.3004 \pm 5 \cdot 10^{-4}$
	TiNi (martensite): ?
	Ti_2Ni: $a = 1.1457 \pm 5 \cdot 10^{-4}$
	Ni_3Ti: $a = 0.5098 \pm 5 \cdot 10^{-4}$ $c = 0.8280 \pm 5 \cdot 10^{-4}$ $c/a = 1.6241$

Table 2

Microstructural parameters of phases

Treatment type	FWHM (degree)		
	TiNi (austenite) (110)	Ni_3Ti (202)	Ti_2Ni (511)
Initial state	0.300 ± 0.025 $(3.4 \cdot 10^{11})$ cm^{-2}	0.084 ± 0.025 $(0.3 \cdot 10^{11})$ cm^{-2}	0.726 ± 0.025 $(20.2 \cdot 10^{11})$ cm^{-2}
Mechanical treatment	0.296 ± 0.025 $(3.3 \cdot 10^{11})$ cm^{-2}	0,185 ± 0,025 $(1.3 \cdot 10^{11})$ cm^{-2}	0,917 ± 0,025 $(32.6 \cdot 10^{11})$ cm^{-2}
Mechanical treatment with pre-hydrogenation for 90 minutes	0.314 ± 0.025 $(3.7 \cdot 10^{11})$ cm^{-2}	0.260 ± 0.025 $(2.5 \cdot 10^{11})$ cm^{-2}	0,904 ± 0,025 $(31.2 \cdot 10^{11})$ cm^{-2}
Mechanical treatment with pre-hydrogenation for 180 minutes	0.330 ± 0.025 $(4.1 \cdot 10^{11})$ cm^{-2}	0.143 ± 0.025 $(0.8 \cdot 10^{11})$ cm^{-2}	0.690 ± 0.025 $(18.0 \cdot 10^{11})$ cm^{-2}

An insignificant decrease in the defect density of powders hydrogenated for up to 180 minutes is apparently due to hydrogen enrichment of defects, which decreases the dislocation density according to [32–34].

Conclusions

The study showed the following:

1. During mechanical treatment of the powder hydrogenated for 180 minutes, the average particle size of titanium nickelide powder decreases almost twice, while the average particle size of the initial powder hardly changes during mechanical treatment.

2. The lattice parameters of the TiNi (austenite), Ti_2Ni, and Ni_3Ti phases in the powder do not change after mechanical treatment and are in good agreement with the literature data. After mechanical treatment of the hydrogenated powders, only the lattice parameter of the Ti_2Ni phase increases and its value is close to the lattice parameter of hydride with $Ti_2NiH_{0.5}$ stoichiometry.

3. The estimated dislocation density in the Ti_2Ni phase is almost an order of magnitude higher than that in TiNi and Ni_3T phases.

Thus, pre-hydrogenation can be effective for powder grinding due to formation of brittle hydride and prevention of fine particle aggregation during high-intensity mechanical treatment.

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Conflicts of Interest

The authors declare no conflict of interest.