Research Article

Comparative Study on Cold Compaction Behavior of TiH_2 Powder and HDH-Ti Powder

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The compaction mechanism of titanium hydride powder is an important issue because it has a direct impact on density and strength of green compacts and ultimately on the physical and mechanical properties of a final sintered products. In this paper, the characteristics and compaction behavior of titanium hydride and hydrogenation-dehydrogenation titanium powders are comparatively studied and analyzed for better understanding of compaction mechanism of brittle low-strength titanium hydride. The results indicate that the particles of titanium hydride powder are easily crushed under compaction loading at relatively low pressure well below compression strength of bulk titanium hydride, the degree of particle crushed increases with the increase of pressure. The compaction behavior of titanium hydride powder mainly includes the rearrangement and crushing of particles in the early compaction stage, minor plastic deformation, if any, and further rearrangement of particle fragments with filling the pores in the later stage. Such compaction behavior provides relative density of green hydride compacts higher than that for titanium powder of the same size. The relatively coarse titanium hydride powder with wide particle size distribution is easier to fill the pores providing highest green density.

1. Introduction

As one of the important metal materials in civil industry and national defense industry, titanium and titanium alloys have a series of excellent properties, such as low density, good welding performance, high specific strength and stiffness, no magnetism, excellent creep and fatigue strength, good corrosion resistance and heat resistance, enough fracture toughness, and excellent biocompatibility, which are widely used in aerospace industry, shipbuilding, marine engineering, petrochemical, automotive, armor, and biomedical fields [1–4]. However, the relatively high cost of titanium limits its application in some fields, first of all, in automotive industry [5, 6]. At present, the powder metallurgy (PM) is an ideal process for producing high-performance titanium alloys at reduced cost, which has a good development

prospect. The simplest and, hence, cheapest powder metallurgy process consists of cold compaction and vacuum sintering of powders or powder blends [7]. Some powder metallurgy approaches use titanium hydride (TiH_2) powder as starting material instead of conventional titanium powders. Such substitution brings a number of processing benefits and improved characteristics of final titanium-based products [8–12]. For such approach, atomic hydrogen evacuated from TiH_2 during vacuum heating, simultaneously cleans the material with taking away the O, C, and Cl impurities. Furthermore, hydrogen release from material promotes the activated sintering via improved surface activity and increased density of crystal lattice defects in dehydrogenated titanium powders. The use of TiH_2 powder has also some economic and technological advantages in the powder metallurgy route.

In the production of alloys and parts with powder metallurgy, powder compaction is an extremely important step to provide not only desirable size and shape but also density and mechanical characteristics of final sintered products. The microstructure, relative density (in other words, porosity), and strength of green powder compacts affect the further densification processes and has an important impact on the microstructure and strength/ductile property balance of final sintered materials [13–16]. For this reason, the cold compaction ability of titanium powders and the relationships between characteristics of green titanium compacts and their densification behavior have become the important subject of numerous studies. Many research articles have reported on the compressibility and the theoretical analysis of the cold compaction behavior of titanium powders using existing or developed compaction equations [17–21]. However, most of the research on the cold compaction was focused on the ductile titanium powder which is similar to that for other ductile metal particles, with relatively high contribution of plastic deformation mechanism to the green density. Compaction of brittle and low-strength TiH_2 powder has not achieved sufficient attention in the research activity [9].

To such powders, an equation that describes the quantitative relationship between the green density of the die-pressed compacts and the applied pressure during the powder compaction has been established [22]. Contrary to ductile metals, the compaction behavior of brittle TiH_2 powder is similar to that for ceramic materials, based mainly on particle rearrangement, crushing, and fragmentation mechanisms, with relatively low, if any plastic deformation contribution [23, 24]. Although there were some studies on the mathematical modelling of TiH_2 powder compaction, such modelling only predicts the trend of compaction behavior, and the analysis of the compaction behavior of TiH_2 powder is not deep and complete [15, 25, 26]. In order to be able to truly reflect the compaction characteristics of TiH_2 powder, this study will analyze it through more systematic analysis methods.

In the current powder metallurgy production, there are many powder forming methods, and the ordinary die-pressing method is still being widely used [27]. The aim of this study was the detailed study of the compaction behavior of TiH_2 powder in the process of uniaxial cold die compaction. Our research was based on measuring the particle size distribution (PSD) and its change under the influence of compacting pressure and analyzing respective change of the TiH_2 compact density. To see the difference, titanium powder produced with hydrogenation-dehydrogenation method (Ti HDH) was used for comparative experiments. Through the comparative studies of two powders, the compaction behavior of TiH_2 powder could be better understood. In order to better analyze the compaction behavior of TiH_2 powder, besides the relative density, fractional volume V^* parameter has been used. Parameter V^* was introduced by Petroni [25] as that associated with the filling of pores at a giving pressure P (pores reduction rate, i.e., decrease in relative pore volume under pressure P applied):

$$V^* = \frac{V_0 - V}{V_0 - V_\infty}, \quad (1)$$

where V_0 is the initial volume of the green compacts, V is the volume at pressure P , and V_∞ is the volume at infinite pressure.

2. Results and Discussion

2.1. Characterization of Initial Powders. Figure 1 illustrates the PSDs of TiH_2 powder fractions, both wide and narrow ones. As shown in Figures 2(a)–2(c) TiH_2 powders are of irregular and nonspherical shape. For powder fractions with a wide PSDs, namely 0–63, 0–88, and 0–125 μm , $D_v(50)$ values are 28.8, 44.9, and 64.4 μm , respectively, while particles of significantly different sizes are observed in such powders (Figure 2(b)). For powder fractions with a narrow PSD, namely, 0–38, 38–63, 63–88, and 88–125 μm , the $D_v(50)$ values are 19.3, 49.6, 73.6, and 108.0 μm , respectively. The sizes of nearly all particles are close to $D_v(50)$ for powders with a narrow PSD, but there are also a few tiny particles that cannot be separated from the large particles with sieving procedure (Figure 2(c)). Initial $D_v(90)$ size for all sieved powder fractions (Table 1) is higher than the upper mesh cell size due to irregular (not spherical) particle shape. Elongated particles can come through the sieve even if their size in the longitudinal direction is bigger than the cell size. Therefore, some amount of coarser particles which size measured by analyzer significantly exceeds mesh cell size always present for each powder fractions.

Since Ti HDH powders were obtained by dehydrogenation of TiH_2 powders of corresponding size, they have similar to TiH_2 morphologies (compare Figures 2(c) and 2(d) as an example) and PSDs (not shown here).

The apparent density is related to the shape, size, surface roughness, and PSD of the powder (Table 2). It can be seen that the wider the PSD of the powders and the larger the particle size, the higher is their apparent density. It should be noted that although the measured densities of TiH_2 and Ti HDH powders with the same PSDs are different due to the difference in theoretical densities of respective bulk materials, however, their relative densities presented in Table 2 are rather close for the same powder size.

The XRD patterns taken from TiH_2 and Ti HDH powders confirmed their single-phase conditions, δ - TiH_2 and α -HCP Ti, respectively (Figure 3).

As it was supposed earlier [22], mostly from an observation of cracking in the TiH_2 particles under compaction loading, they fracture due to their specific mechanical properties (brittleness and low strength) [17]. It was supposed also that such fracturing lead to a fragmentation of coarse particles what was considered as one of the factors of sintering activation due to opening of the new boundaries serving as vacancies sinks upon pore healing. Similar cracking was observed in this study as well (Figure 4(a)) and as expected, no cracking was found in the Ti HDH particles (Figure 4(b)). However, only now, we were able to quantitatively estimate this fragmentation using the method described in the experimental section. SEM investigation

FIGURE 1: The PSD of the sieved TiH_2 powder fractions. —■—0–38 μm , —●—0–63 μm , —▲—0–88 μm , —◆—0–125 μm , —□—38–63 μm , —○—63–88 μm , and —△—88–125 μm .

FIGURE 2: The SEM micrographs of initial powders: (a) 0–38 μm TiH_2 , (b) 0–125 μm TiH_2 , (c) 88–125 μm TiH_2 , and (d) 88–125 μm HDH Ti.

TABLE 1: Dv (50) and Dv (90) sizes of some starting TiH_2 powders and its change after compaction depending on pressure applied.

	Sieved size (μm)	Initial size (μm)	Size (μm) after compaction with pressure (MPa)								
			250	300	350	400	450	500	550	600	650
Dv (50) (μm)	38–63	49.6	38.0	37.4	34.0	21.0	13.9	13.1	15.3	12.6	12.2
	63–88	73.6	49.9	38.0	40.5	36.9	28.2	29.1	28.6	26.7	23.0
	88–125	108.0	60.9	53.2	42.1	34.5	36.1	31.8	29.2	28.1	18.1
Dv (90) (μm)	38–63	81.9	72.1	70.3	68.0	52.9	38.9	37.7	39.2	47.6	32.9
	63–88	122	98.8	92.2	91.1	96.9	94.8	95.1	87.4	94.0	92.0
	88–125	188	138.0	128.0	93.9	122.0	112.0	117.0	91.9	116.0	114.0

TABLE 2: Apparent relative densities of TiH_2 and HDH titanium powders of various particle sizes, as well as relative densities of corresponding green compacts pressed at 250–650 MPa. Theoretical density of bulk materials was taken as 3.75 g/cm^3 for TiH_2 and 4.51 g/cm^3 for HDH-Ti.

Powder type and size (μm)	Apparent density (g/cm^3)	Relative density									
		Compaction pressure (MPa)									
		250	300	350	400	450	500	550	600	650	
TiH_2	0–38	0.32	0.720	0.760	0.786	0.799	0.822	0.830	0.847	0.858	0.865
	0–63	0.41	0.742	0.762	0.782	0.801	0.819	0.833	0.846	0.861	0.870
	0–88	0.42	0.768	0.787	0.799	0.822	0.838	0.850	0.867	0.879	0.887
	0–125	0.44	0.782	0.805	0.818	0.834	0.847	0.858	0.869	0.883	0.888
	38–63	0.39	0.729	0.748	0.779	0.792	0.807	0.826	0.847	0.856	0.871
	63–88	0.41	0.745	0.768	0.788	0.806	0.825	0.842	0.857	0.863	0.882
	88–125	0.44	0.759	0.777	0.802	0.821	0.834	0.853	0.867	0.874	0.889
Ti-HDH	0–38	0.28	0.651	0.678	0.704	0.727	0.749	0.766	0.783	0.797	0.811
	0–63	0.32	0.668	0.699	0.725	0.750	0.786	0.785	0.802	0.819	0.831
	0–88	0.33	0.677	0.709	0.735	0.757	0.774	0.790	0.810	0.824	0.834
	0–125	0.38	0.700	0.731	0.743	0.774	0.791	0.805	0.824	0.836	0.848
	38–63	0.39	0.654	0.695	0.712	0.734	0.759	0.772	0.789	0.815	0.820
	63–88	0.40	0.674	0.702	0.732	0.755	0.783	0.793	0.811	0.832	0.837
	88–125	0.43	0.692	0.718	0.750	0.773	0.788	0.810	0.821	0.834	0.849

FIGURE 3: XRD patterns of initial powders: (a) TiH_2 δ phase (■) and (b) Ti HDH α phase (●).

FIGURE 4: SEM images showing characteristic difference between green compact structure for (a) TiH_2 and (b) Ti HDH. Compaction pressure 300 MPa.

clearly confirmed the fragmentation (Figure 5) while analyzing the PSDs of the powder fractions after disintegration of the pressured compacts (Figure 6) allowed to obtain a comprehensive set of data on change of powder sizing (Table 1). The results not only gave a direct confirmation of the suggestions made earlier about the TiH_2 fragmentation but also allowed to analyze the fragmentation depending on the initial size of the powder particles and the pressure value. The reduction of particle size, presented in Table 1 was evaluated for both $D_v(50)$ and $D_v(90)$ as $(D(0) - D)/D(0)$, where $D(0)$ is an initial particle size, and D is size after compaction. The reduction of $D_v(50)$ size is huge, being around 70% at high compaction pressures. It is clearly seen from Figure 7 that all fractions have undergone the fragmentation which is less pronounced in the pressure range above 450 MPa (what is confirmed by very similar PSDs obtained for TiH_2 powder compacted at 450 and 600 MPa, Figure 6) and for the finest fraction. Due to that a difference in the average value presented by $D_v(50)$ decreases with pressure and becomes rather small at high pressures. Decrease in $D_v(90)$ value obviously takes place as well but is less pronounced, especially at high pressures, presumably because of possible agglomeration.

To explain the above trends in the dependence of fragmentation on the pressure and the initial powder particle size, one has to take into account that fracturing of the particular powder particle in the powder ensemble is influenced not by the overall compaction pressure. More important is the internal compression force between adjacent powder particles that are generally higher than total compacting (external) pressure. Crushing occurs when local stresses in the contact between particles is higher than compression strength of titanium hydride (250 MPa). At low external pressure, relatively loose packing of powder results in a low number of contacts. The external pressure creates sufficient stresses at the contacts only, thus crushing a limited number of TiH_2 particles. This number is certainly increasing with pressure growth, regardless of the size of initial particles. For all studied powder fractions, the particle size reduces intensively with the increase of pressure up to ~450 MPa. Respective PSDs are shifting to the left and widening (Figure 6). However, at further increase in compaction pressure from 450 to 600 MPa, the rate of particle size reduction slows down (Figure 7); as a result, evolution of the PSDs is not as significant as at lower pressures (Figure 6). Number of particle fragments increases at medium and high pressure values providing their closer packing, that is, number of contacts between particles increases. For such powder ensemble, the crushing forces are less localized and distributed more uniformly over compacts, at the same time, being partially blocked with the higher friction between particles and their fragments. Under such condition, the pressure increase is less effective in the fragmentation.

Size of TiH_2 particles is an important parameter affecting fragmentation. Coarser powders fractions are more inclined for fragmentation at low pressures. For example, at 250 MPa, the $D_v(50)$ size reduction of coarse TiH_2 particles is already 43.6%, while that of fine particles is 23.4% only. The coarsest particles earlier than others are fixed in the position

promoting sufficiently high local stresses and creating “skeleton” for transfer of compression loading between particles. Therefore, the coarsest particles are easy to be crushed at low pressure. As pressure grows, more and more powder particles of all sizes are inclined for fragmentation and the influence of initial particle size on fragmentation is gradually decreasing. When the pressure is high enough, the crushing degree of coarse and fine particles is roughly the same.

2.2. Comparative Compaction Behavior of the Powders.

The process of powder compaction can be generally divided into three overlapping stages: rearrangement of particles, their elastic and plastic deformation, and fracture or fragmentation. Relative input of each stage in the compact densification strongly depends on the powder particle characteristics, such as their size, shape, roughness of the surface, and its atomic structure, mechanical properties (strength and ductility). The binding of particles determines the strength of the compacts, which includes both mechanical locking caused by displacement and deformation of the surface during compaction, as well as the interaction forces between atoms on the surface of adjacent particles. The first is more relevant to the particles with irregular shapes and rough surfaces, while the second gives main contribution when atoms on the surfaces of adjacent particles become close to each other at the late stages of powder compaction. It is quite naturally expected that powders with different plastic deformation abilities will behave in different way upon compaction. All the data on the change of relative densities achieved by different pressures upon compaction of both TiH_2 and Ti HDH powders are presented in Table 2 and will be further analyzed in Figures 8 to 13.

As it was shown in the previous section, TiH_2 powder was fragmented even under relatively low compaction pressure. As expected, the densities of all TiH_2 fractions strongly depend on the pressure (Figure 8(a)), but the rate of density increase becomes slightly lower at higher pressure. However, not only the pressure but the powder size as well defines the compact density. For the same pressure, the density is always highest for the coarsest fraction and, taking into account the results presented in the previous section, one can assume that this is due to the more effective fragmentation of the bigger size powder particles. In line with such explanation is the result that an influence of the powder sizing is decreasing with the pressure increase because at high pressures, PSDs of the initially different fractions becomes much extent the same. Such trend is true for both narrow and wide fractions but is more pronounced for the wide ones. Comparison of the narrow (88–125 μm) and wide (0–125 μm) fractions with the same maximum size clearly shows that presence of very fine particles expedites densification, presumably due to more effective rearrangement leading to the filling of the initial pores. For the fractions with the smaller than 125 μm maximum size, the effect of fine particles is not obvious. If it is assumed that the initial porosity of the coarser fractions is also coarser and is

FIGURE 5: SEM images of TiH₂ powder: (a) initial particles (88–125 μm) without compaction and particles after compaction at (b) 300 MPa, (c) 450 MPa, and (d) 600 MPa.

FIGURE 6: Continued.

FIGURE 6: The influence of different compaction pressures on PSD of initial TiH_2 powder: (a) 38–63 μm TiH_2 , (b) 63–88 μm TiH_2 , and (c) 88–125 μm TiH_2 . —■—initial powder, —●—powder after compaction (300 MPa), —▲—powder after compaction (450 MPa), and —◆—powder after compaction (600 MPa).

FIGURE 7: Decrease in average particle size of TiH_2 powder after compaction depending on pressure. —■—38–63 μm , —●—63–88 μm , and —▲—88–125 μm .

FIGURE 8: The green densities versus compaction pressure for (a) TiH_2 and (b) Ti HDH powders of various PSDs. ■—0–38 μm , ●—0–63 μm , ▲—0–88 μm , ▼—0–125 μm , ◆—38–63 μm , ◀—63–88 μm , and ▶—88–125 μm .

FIGURE 9: Relative densities (a–d) and V^* parameters (e–h) versus compaction pressure for TiH_2 powder and HDH titanium powder. ■— TiH_2 38–63 μm , ●— TiH_2 88–125 μm , ▲— TiH_2 0–63 μm , ▼— TiH_2 0–125 μm , □—HDH-Ti 38–63 μm , ○—HDH-Ti 88–125 μm , △—HDH-Ti 0–63 μm , and ▽—HDH-Ti 0–125 μm .

FIGURE 10: SEM of green compacts: (a) TiH₂ 300 MPa, (b) TiH₂ 800 MPa, (c) TiH₂ 800 MPa (high multiple), (d) HDH titanium 300 MPa, (e) HDH titanium 800 MPa, and (f and g) HDH titanium 800 MPa (high multiple).

FIGURE 11: SEM images of TiH_2 green compacts: (a) 0–38 μm 250 MPa, (b) 0–38 μm 650 MPa, (c) 0–125 μm 250 MPa, (d) 0–125 μm 650 MPa, (e) 88–125 μm 250 MPa, and (f) 88–125 μm 650 MPa.

FIGURE 12: SEM images of HDH titanium green compacts: (a) 0–38 μm 250 MPa, (b) 0–38 μm 650 MPa, (c) 0–125 μm 250 MPa, (d) 0–125 μm 650 MPa, (e) 88–125 μm 250 MPa, and (f) 88–125 μm 650 MPa.

FIGURE 13: Comparison of relative density (a) and V^* parameter (b) versus pressure for coarse TiH_2 and titanium powders as a typical example of different compaction behavior for these materials. ■— TiH_2 and ●—HDH-Ti.

easier to be filled by the rearrangement of the fine particles, this can be understood.

Densification of the Ti HDH powders generally occurs following similar to the TiH_2 dependencies on the pressure and powder sizing, that is, the coarser the powder and the higher the pressure, the higher density is achieved (Figure 8(b)). The main difference is that relative density values achieved in Ti HDH for the same fraction sizes and the same pressures are lower than those for TiH_2 , as it is seen from Figure 9. Two more specific features of the Ti HDH powder compared to that of the TiH_2 powder should be mentioned. First, addition of fine particles to the starting powder, being effective for the TiH_2 , does not improve the densification of Ti HDH; therefore, narrow and wide fractions of the same maximum size exhibit identical response to the pressure increase (Figure 9). Second, for the Ti HDH powders, not as for TiH_2 powders, in which the difference in densities for coarse and fine titanium powders is obviously reduced with pressure increase, it remains nearly constant over the pressure range studied.

A difference in the compaction behavior of the two powder types is also confirmed by SEM observations (Figure 10). As shown in Figures 10(c) and 10(f), the compacted TiH_2 powder particles do not demonstrate any indications of the plastic deformation even at high pressure, while traces of such deformation on the surface of Ti HDH particles confirm its considerable contribution into the densification. The above difference is reflected not only in the powder particle size and structure but in the morphology of the pore system. It can be seen from Figures 11 and 12 that under the same conditions, the total volume fraction and size of pores in the TiH_2 green compacts are noticeably lower than that in the Ti HDH compacts. Pores in the TiH_2 powder are filled with small crushed particles, thus their size and, of course, total volume that define the density are relatively small even at low compaction pressure of 250 MPa, as shown in Figure 11(a), 11(c), and 11(e). The total volume fraction of pores is noticeably reduced with increase in compaction pressure due to more and

more fragments filling the space between the crushed particles (Figure 11). Other features of the porosity are observed in the Ti HDH powder (Figure 12), in which the size and total volume of pores is larger, compared to TiH_2 case for all studied pressures, especially for coarse powder particles. Although voids between titanium particles forming a starting porosity system are reduced at higher pressures due to particle plastic deformation, thus resulting in corresponding density increase, such evolution of porosity is less effective than that in the TiH_2 powder. In accordance with results above, the density and the V^* parameter for TiH_2 powder are always higher than for Ti HDH powder for the same particle size within all studied pressure range (Figure 9).

Thus, one can conclude that both, TiH_2 and Ti HDH powders can be effectively densified by the compaction load, but surprisingly, the first exhibits a higher ability for the densification at equivalent parameters: powder sizing and pressure. This result certainly shows that densification mechanism by fragmentation and mechanical locking between the particles of irregular shape and/or their fragments formed under the compaction load, without very little, if any plastic deformation is not only possible but a very effective in producing green preforms from the TiH_2 powder, quite strong and suitable for further handling.

An explanation can be found using the V^* value, which is a characteristic of the porosity system, and its change is more relevant to analyze the rate of the porosity healing than the change of the density. The difference is clearly seen if one compares Figure 9(a)–9(d) with 9(e)–9(h). V^* parameter is usually higher for TiH_2 within the whole studied pressure range, illustrating more complete filling the pores in titanium hydride compacts. Moreover, change of the V^* parameter clearly demonstrates a difference between processes supporting the densification of the narrow and wide fractions of the coarse Ti HDH powder. Due to the availability of the fine particles and their ability to fill in the empty places (i.e. big pores) in the 0 to 125 fraction, the filling of the porosity proceeds much effective than in the 88 to 125

fraction, especially at a relatively low pressure although the relative density does not show this difference between the two fractions (Figure 9(c) and 9(g)).

One of the study results on the densification of the powders presented earlier is that even low compaction pressure, such as 250 MPa, has already been capable of reaching quite a reasonable green density (Figure 8). This result is important per se because in many practices, using high pressures is technically impossible, which is especially true for big size parts or semiproducts, slabs, or billets produced by cold isostatic pressing (CIP) technique. Therefore, it was interesting to understand how the powders respond to even lower than 250 MPa pressures. For that, the coarsest fraction 88 to 125 μm of both type powders were exposed to the pressures in the 100 to 250 MPa range. This particular fraction was selected to minimize a role of fine particles that would definitely actively participate in the densification by their rearrangement and to elucidate the compaction mechanism of the relatively big particles and, especially, to estimate what powder is more suitable for a low-pressure compaction. The density change of both powders is presented in Figure 13(a) starting from their apparent densities; corresponding change of the V^* value is shown in Figure 13(b). An advantage of the TiH_2 powder looks convincing. Not excluding the input of the rearrangement of starting size particles, probably equal for both powders, one can definitely state that densification through the fragmentation of the TiH_2 particles works more effectively than plastic deformation of the Ti HDH particles, thus providing intensive filling of the porosity. It is important that such advantage of the TiH_2 powder achieved at low pressures remains valid in the whole pressure range (Figure 8).

The results of this study prove that coarse TiH_2 powder with wide PSD is preferable to achieve high relative density of green compacts. However, high density of green compacts is desirable but not sufficient to ensure achievement of highest density during further sintering. It is well known that coarse powders usually demonstrate weaker sintering ability than fine ones. Having in mind attainment of maximal possible density of sintered titanium products, which is important for admissible mechanical characteristics, further investigation should be performed to clarify influence of titanium hydride powder size on microstructure evolution of powder compacts and pore healing during sintering.

3. Conclusion

The expressed brittleness and low strength of TiH_2 powder are its distinctive features affecting compaction behavior and providing achievement of higher relative density of compacts as compared to highly ductile HDH titanium powder. The particles of TiH_2 powder are easily crushed during the compaction process at pressures well below compressive strength of bulk titanium hydride. At low pressure, coarse TiH_2 powder is more inclined to be crushed than fine TiH_2 powder. At high pressure, the crushing is less affected with particle size.

The crushing and rearrangement of TiH_2 particles and their fragments makes the main contribution to the densification of the green compacts while contribution of plastic

deformation is minor. At pressure rise, the more and more TiH_2 particles are crushed with rearrangement of powder fragments promoting pore filling and, hence, higher density of green compacts. The particle crushing with rearrangement of fine fragments resulted in formation of fine pores within wide pressure range, while size of starting powders does not noticeably affect pore size in green compacts.

The relatively coarse TiH_2 powder with wide PSD (0–125 μm as an example) is suitable for better pore filling, so such powder can be recommended to achieve high density of green compacts.

4. Experimental Section

TiH_2 powder was obtained by hydrogenation and ball milling of titanium sponge. In the process of hydrogenation, titanium sponge was heated under vacuum and ultrapure hydrogen was introduced into the vacuum sintering furnace (SIOMM) at 550°C until the hydrogenation process was completed. After that the hydrogenated titanium sponge was milled by planetary ball mill (QM-2SP20-CL) to obtain TiH_2 powder which was then sieved to obtain size fractions with wide (0–63, 0–88, and 0–125 μm) or narrow (38–63, 63–88, and 88–125 μm) PSD. Fraction of 0 to 38 μm was also obtained, which will be further considered as a representative of both wide and narrow PSDs. In the majority previous researches and industrial productions, the powders with wide PSD are being usually used, in which initially small particles mask crushing of larger particles upon compaction. Therefore, the use of narrow PSD powder was considered as useful to carry out comparative experiments on compaction behavior of TiH_2 powders of different sizes. The PSDs for all selected size fractions were measured using a Malvern Mastersizer 3000 laser instrument (Figure 1). Taken from PSDs, the $D_v(50)$ size values below which is 50% of powder material and 50% of material is above, were used to represent the average particle size. Also, $D_v(90)$ size values below 90% of the powder material were included in the analysis to characterize the coarsest particles (Table 1).

Ti HDH powders of the same sizes were obtained by dehydrogenation of the corresponding fractions of TiH_2 powder. Figure 2 illustrates the scanning electron microscopy (SEM) micrographs of different types and particle sizes of the initial powders.

The apparent (loose) densities of initial powders were determined using the graduated cylinder approach in accordance with the standard test method [28].

Four-column hydraulic press (HJS32-100) was used for die compaction of powders. Green powder compacts of the fixed mass (25 g) of rectangular shape (length of 65 mm and a width of 10 mm) were uniaxially pressed using different pressure values. Powder compaction was performed without using of any lubricant, but the die walls were cleaned with absolute ethanol before pressing.

The density of green compacts ρ was calculated using their sizes and mass. The relative density values were determined as the ρ/ρ_0 ratio, where ρ is measured density, ρ_0 is the theoretical density of the corresponding bulk material, taken 4.51 g/cm³ for titanium and 3.75 g/cm³ for TiH_2 .

In order to study the fragmentation of TiH_2 powder upon cold compaction and for better understanding of compaction mechanism, the PSD of TiH_2 particles before and after compaction were comparatively measured. The specially developed procedure was used to study PSD of TiH_2 fragments formed upon compaction. The green compacts were heated to 99°C using a constant temperature water bath (HWS-24) and hold in heated condition for 2 hours. After that, the heated green compacts were put into an ultrasonic vibration device (GT Sonic-D6) for compact disintegration into fragments under vibration influence. Finally, the PSD of the obtained disintegrated fragments was measured by the wet method of Malvern Mastersizer 3000 device. SEM was used for analyzing starting powders and their evolution upon compaction.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

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