

Article

T15 High Speed Steels Produced by High-Temperature Low-Pressure Short-Time Vacuum Hot-Pressing Combined with Subsequent Diffusion-Bonding Treatment

Wentao Shan and Yaojun Lin *

School of Materials Science and Engineering, Wuhan University of Technology, Wuhan 430070, China; shan18438621709@163.com

* Correspondence: yjlin@whut.edu.cn; Tel.: +86-13971439387

Abstract: Currently, hot isostatic pressing (HIP) is widely used to produce highly alloyed high speed steels (HSSs) in an industrial scale; however, the HIP's production cost is very high. Another powder consolidation approach with low production cost, namely vacuum hot-pressing (VHP), has hitherto received limited attention. The present work aims to develop an innovative solid-state VHP approach, producing HSSs with large cross-sectional sizes via a VHP facility having low loading capacity, thus further decreasing production cost. In doing so, VHP is performed at a sufficiently high temperature such that the pressure leading to full densification can be significantly reduced to a magnitude as low as several MPa; simultaneously, VHP is completed within a timeframe as short as several seconds to minutes, retaining fine carbide sizes; subsequently, the as-VHP HSS is diffusion-bonding treated (DBT-ed) at a relatively low temperature, achieving full metallurgical bond between powders while minimizing carbide growth. In the present work, T15 HSS was processed using the above VHP approach. The VHP temperature as high as 1200 °C was selected and consequently, the minimal pressure leading to full densification was decreased to ~7 MPa. By controlling displacement of pressing punch to a value corresponding to full densification, the VHP was completed for only 15 min. The almost fully dense as-VHP T15 HSS exhibits submicrometric carbide sizes smaller than those in the as-HIP counterpart, but incomplete metallurgical bond between powders. After diffusion bonding treatment at a relatively low temperature of 1100 °C for 2–4 h, the extent of metallurgical bond between powders is significantly enhanced with insignificant carbide growth. After regular quenching and tempering, the VHP plus DBT-ed T15 HSSs exhibit smaller average primary carbide sizes and similar hardness and three-point bend fracture strength, relative to those in the HIP counterpart after similar quenching and tempering.

Keywords: high speed steel; powder metallurgy; vacuum hot-pressing; hardness; bend fracture strength



Citation: Shan, W.; Lin, Y. T15 High Speed Steels Produced by High-Temperature Low-Pressure Short-Time Vacuum Hot-Pressing Combined with Subsequent Diffusion-Bonding Treatment. *Metals* **2023**, *13*, 998. <https://doi.org/10.3390/met13050998>

Academic Editor: Filippo Berto

Received: 19 April 2023

Revised: 14 May 2023

Accepted: 19 May 2023

Published: 21 May 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

High speed steels (HSSs) is a class of tool steels containing high contents of carbon and alloying elements, including W, Mo, Cr, V, Co, etc. They exhibit high hardness and wear resistance at room temperature and even at elevated temperature [1]. As a result, they are widely used to manufacture cutting tools, as well as dies and wear-resistant parts [2–4]. It is well established that size, distribution, morphology, and amount of primary carbides critically affect mechanical properties of HSSs [5,6]. Fine carbides uniformly dispersed in the matrix can significantly improve the performance and service life of the high-speed steels [7–9]. During conventional casting, slow cooling rate together with high contents of C and alloying elements leads to coarse ledeburite eutectic structure and severe carbide segregation [10–12], which promote nucleation and quick propagation of microcracks along coarse carbides, and thus early failure of as-cast HSSs [13]. Although the coarse herringbone

carbide can be broken in the subsequent hot-working process, the coarse granular carbides and residual segregation will still significantly deteriorate the mechanical properties of HSS.

In order to solve the above problems, a large number of powder metallurgy (PM) techniques have been developed, such as hot isostatic pressing (HIP), supersolidus liquid phase sintering (SLPS), spark plasma sintering (SPS), additive manufacturing (AM), etc. These techniques usually use inert gas or water atomized prealloyed powders as raw materials. The ultra-high cooling rates during atomization process eliminate segregation and produce fine and uniformly distributed carbides [14–16]. However, the above PM techniques have their advantages and disadvantages. Although SLPS can make large-size fully dense HSSs, high sintering temperature inevitably leads to carbide coarsening [17]. In addition, pressureless SLPS requires compacts with sufficiently high green strength as precursors. During cold pressing of water atomized powders, shaping agents need to be added. Difficulty to completely remove shaping agents during SLPS introduces additional oxygen and other impurities [18,19]. SPS can produce fully dense HSSs at lower temperatures and for shorter time than conventional powder consolidation techniques, but the size of products that can be produced by SPS is small [20,21]. As an emerging PM technology, AM manufacturing has the advantage of rapid solidification, yielding fine carbides [22,23]. It can also be used to manufacture dies and molds with complex geometry [24]. However, low productivity of AM and high susceptibility of AM processed HSSs to cracking limit widespread applications of AM in large-scale production of HSSs [25]. As a widely used PM technique to produce HSSs in industrial scale, HIP enables full densification at lower temperatures, retaining fine carbides [16,26,27]. However, very expensive equipment and complicated multiple processing steps result in high production cost during HIP. Another well-developed PM technique, namely vacuum hot pressing (VHP), is also able to consolidate HSS powders to full densification at lower temperatures and thus to achieve the retention of fine carbides. Interestingly, VHP can also produce HSSs at the industrial scale, but with much lower production cost than that during HIP. However, VHP has heretofore received limited attention [19,28].

In a related study [28], Qiu et al. consolidated prealloyed powders of a high C and Cr tool steel Fe-2.6C-2.3V-1.1Mo-1.4Nb (wt%) by VHP at temperatures of 1100 and 1150 °C (far below the solidus temperature of 1247 °C) for 2 h under 40 MPa, obtaining almost fully dense tool steel. After quenching at 1150 °C and double tempering at 500 °C for 2 h, the tool steel exhibits average carbide sizes of 3.5 and 5.5 µm, maximum carbide sizes of 6.0 and 8.5 µm, hardness 62.6 and 60.8 HRC, and three-point bend fracture strength of 2060 and 1850 MPa, respectively. In another related study [19], Wang et al. consolidated M4 HSS prealloyed powders at 1220–1230 °C close to the solidus temperature of 1240 °C. As a result, the pressure leading to full densification can be reduced to a value as low as 4.5 MPa. As the authors suggested, the very low pressure enables the production of HSSs with large cross-sectional sizes using a VHP machine having low loading capacity, which further reduces the production cost. However, although nearly full densification (relative density of 99.85%) was achieved with a holding time of 50 min, carbides coarsen to the sizes ranging from 3 to 10 µm due to prolonged holding time at the very high temperature.

In view of the drawback involved in the high-temperature VHP process as reported in Ref. [19], the objective of the present work is to develop an innovative high-temperature low-pressure short-time solid-state VHP process. In the present work, short-time leading to full densification is attained by monitoring the displacement of the pressing punch. Once the displacement reaches the value corresponding to full densification, the heating and pressing are immediately terminated to end the VHP process. By doing so, unnecessary holding time is saved. Moreover, the low resistance of the very high temperature powders to the pressing punch movement enables the high speed of the punch movement, thus shortening time of the punch movement from the starting position to the position corresponding to full densification. Given that VHP in the present work is a solid-state consolidation process and involves slight shear deformation between powders, mutual diffusion of elements at powder boundaries should be the predominant mechanism responsible for the formation of

metallurgical bond between powders. The short time during VHP may lead to incomplete metallurgical bond between powders. Therefore, diffusion-bonding treatment (DBT) at a relatively low temperature is applied to the as-VHP HSS, attaining complete metallurgical bond between powders while minimizing carbide growth. Selecting T15 HSS as a model material, the microstructure and mechanical properties (specifically hardness and three-point bend fracture strength) in as-VHP T15 HSS and VHP + diffusion-bonding treated (DBT-ed) T15 HSSs were investigated and compared with those in the HIP counterpart. The selection of T15 HSS as a model material is due to availability of microstructures and mechanical properties of HIP T15 HSS in the published literature [16,26], allowing the comparison of microstructures and mechanical properties between VHP T15 HSS in the present work and HIP T15 HSS.

2. Materials and Methods

2.1. Characterization of T15 Prealloyed Powders

T15 steel prealloyed powders used in the present work were produced using high-pressure nitrogen atomization by Xingyuan Powder Metallurgy Company of Hunan Institute of Metallurgical Materials. The powder size was measured by Mastersizer 2000 laser particle-size analyzer. The chemical composition of the powders was measured by chemical analysis using (1) inductively coupled plasma atomic emission spectroscopy for metallic elements, (2) spectrophotometric method (Chinese GB/T 7729-2021) for Si, (3) infrared absorption method after combustion in an induction furnace (Chinese GB/T 20123-2006) for C, (4) pulse heating inert gas fusion infrared absorption method (Chinese GB/T 11261-2006) for O, and (5) thermal conductimetric method after fusion in a current of inert gas (Chinese GB/T 20124-2006) for N. The morphology of the powder was observed by a ZEISS Gemini 300 field emission gun (FEG) scanning electron microscope (SEM).

Before powder consolidation, powders of 120.67 g were loaded into the graphite mold having an internal alumina tube (to prevent carburization) with inner diameter of 40 mm. The height of as-loaded powders is 18.85 mm at room temperature. The height of the fully dense T15 HSS was calculated to be 11.64 mm at temperature with theoretical density of 8.25 g/cm^3 [29]. By considering the effect of thermal expansion [30], the as-loaded and fully dense heights were calculated to be 19.12 and 11.80 mm, respectively, at the target VHP temperature of 1200 °C. The graphite mold with powders was moved into the VHP facility's chamber, and the chamber was evacuated to a pressure of $4.0 \times 10^{-2} \text{ Pa}$. A uniaxial pressure of 7 MPa (the minimal pressure that the VHP facility can provide) was applied to the powders, and the powders were heated to the target temperature of 1200 °C at the heating rate of 10 °C/min. During heating, the displacement of the pressing punch was monitored. The experimental observation found that, during heating to 1200 °C, although the pressing punch moved, its displacement did not reach the value corresponding to full densification. When holding at 1200 °C, the pressing punch continued to move, and took 15 min to the position corresponding to full densification. Once the pressing punch reached the position corresponding to full densification, heating and pressing were immediately terminated to end the VHP process.

Subsequently, the as-VHP T15 HSS was DBT-ed at 1100 °C for 2 and 4 h, respectively, in vacuum without the application of pressure. After DBT, the T15 HSSs cooled to room temperature in the furnace. For convenience, VHP + DBT-ed T15 HSSs at 1100 °C for 2 and 4 h are designated as VHP + DBT-ed@1100°C_2h and VHP + DBT-ed@1100°C_4h T15 HSSs, respectively. VHP, VHP + DBT-ed@1100°C_2h and VHP + DBT-ed@1100°C_4h T15 HSSs were quenched by austenitizing in vacuum at 1204 °C for 5 min, then oil quenching, and tempered at 550 °C for 2 h for 3 times in argon atmosphere.

2.2. Phase and Microstructure Analysis of the Consolidated T15

X-ray diffraction (XRD) analysis was performed in Bruker D8 Advance X-ray diffractometer with Cu K α radiation (wavelength $\lambda = 1.5406 \text{ \AA}$) and with the scanning range of 20–100° and the scanning speed of 0.4°/min, in order to identify the phases in the as-VHP

and VHP + DBT-ed T15 HSSs. Microstructures in the as-VHP and VHP + DBT-ed T15 HSSs were characterized by a ZEISS Gemini 300 FEG SEM equipped with energy-dispersive X-ray spectroscopy (EDS) at acceleration voltage of 15 kV. SEM specimens were prepared by standard metallographic technique and microstructures were revealed by an etchant of 4% nitric acid in alcohol. Based on SEM BSE micrographs, carbide sizes were measured on approximately 500 carbides in randomly selected regions using Image-Pro Plus software, obtaining carbide size statistical distribution and volume fraction. By fitting the carbide size statistical distribution with a lognormal probability function, average carbide size ($\bar{d}$) and standard deviations (d_1 and d_2) were computed: $d = \bar{d} \cdot \frac{d_1}{d_2}$.

2.3. Density Measurement of the Consolidated T15

The samples of as-VHP and VHP + DBT-ed T15 HSSs were cut and then ground and polished. The densities were measured using Archimede's method.

2.4. Mechanical Property Testing of the Consolidated T15

For the as-VHP and VHP + DBT-ed T15 HSSs after quenching and tempering, Rockwell HRC hardness was measured at 20 randomly selected points on polished surfaces using a Huayin 200HRS-150 digital Rockwell hardness tester. An Instron 5966 universal tester was used to measure the three-point bend fracture strength of the as-VHP and VHP + DBT-ed T15 HSSs after quenching and tempering at room temperature. The specimens for three-point bend testing were sectioned into the following dimensions: $25 (\pm 1 \text{ mm}) \times 4 (\pm 0.5 \text{ mm}) \times 2 (\pm 0.5 \text{ mm})$, by electric discharge machining, and polished on each surface. During three-point bend testing, the span for testing is 16 mm, and the bending rate is 0.1 mm/min. For consistence of results, five specimens for each type of the T15 HSSs were tested. After three-point bend testing, the fractured surfaces were observed with the above ZEISS Gemini 300 FEG SEM.

3. Results

3.1. Composition and Morphology of T15 Prealloyed Powders

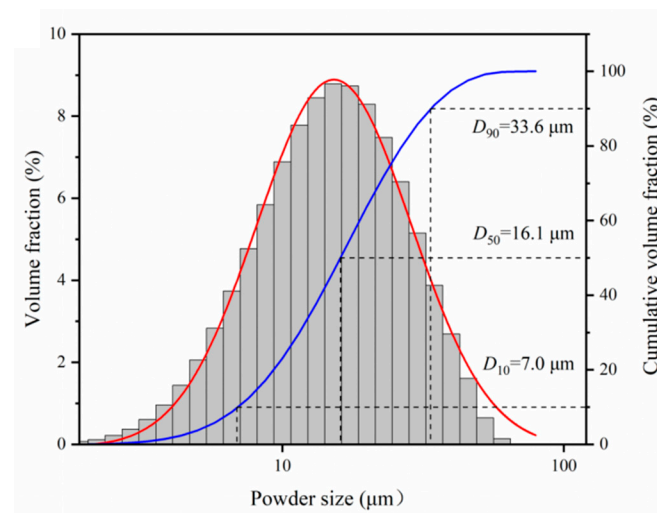
The measurement results are shown in Table 1, indicating very low oxygen and nitrogen contents. The powder size distribution was presented in Figure 1a, with D_{10} , D_{50} , and D_{90} being 7.0, 16.1, and 33.6 μm , respectively. The powder morphology is demonstrated in Figure 1b,c, showing spherical morphology for most powders and occasionally observed satellite and rod powders.

Table 1. Chemical compositions of prealloyed T15 powders (wt.%).

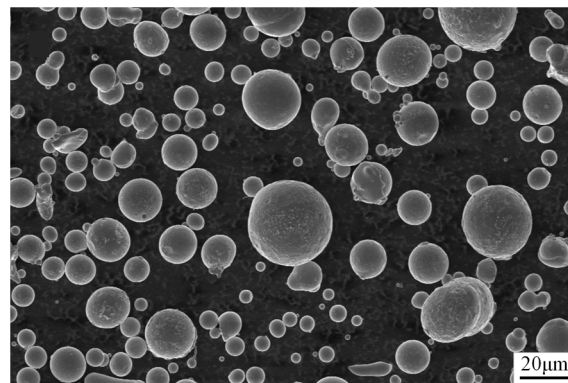
C	Si	Mn	Cr	W	Mo	V	Co	O	N	Fe
1.67	1.02	0.45	5.12	12.66	0.24	5.18	5.29	0.030	0.097	Balance

3.2. XRD Results

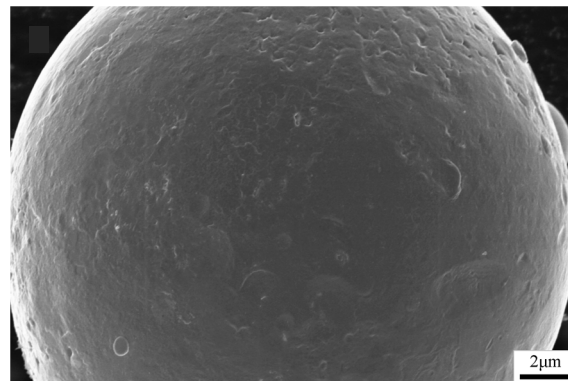
Figure 2 shows the XRD patterns of as-VHP and VHP + DBT-ed T15 HSSs before and after quenching and tempering. Before quenching and tempering, the XRD patterns of as-VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h contain diffraction peaks of α -Fe, $\text{Fe}_3\text{W}_3\text{C}$ (M_6C type carbide), and VC (MC type carbide). Due to slow cooling rates after VHP and DBT, α -Fe may be pearlitic ferrite or pearlitic ferrite together with very low amount of martensite and bainitic ferrite. After quenching and tempering, the XRD patterns of as-VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h still include the diffraction peaks of α -Fe, $\text{Fe}_3\text{W}_3\text{C}$ (M_6C type carbide), and VC (MC type carbide). Unlike as-VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h before quenching and tempering, α -Fe should be tempered martensite after quenching and tempering.



(a)



(b)



(c)

Figure 1. Characteristics of the prealloyed T15 powders: (a) powder size distribution, (b) powder morphology, and (c) higher magnification powder morphology showing surface topograph.

3.3. Microstructures in VHP T15 HSSs

Figure 3a shows the SEM BSE microstructure of as-VHP T15 HSS. The brighter white carbides and darker gray carbides are uniformly distributed in the as-VHP T15 matrix. According to EDS analysis, the brighter white carbides are W-rich M_6C carbides, and the darker gray carbides are V-rich MC-type carbides. The size statistical distributions of M_6C , MC, and M_6C+MC carbides, as well as the corresponding fitted lognormal distribution curves, in the as-VHP T15, are shown in Figure 3b, and carbides sizes are determined as $d = 0.59^{+0.11}_{-0.09}$, $0.38^{+0.05}_{-0.04}$ and $0.48^{+0.09}_{-0.07}$ μm for M_6C , MC, and M_6C+MC carbides, respectively. The average size of M_6C+MC carbides is smaller than those in the as-HIP T15 [16]: 1.0 μm

for HIP at 1130 °C for 4 h; 1.2 μm for HIP at 1195 °C for 4 h. The volume fractions of M_6C and MC carbides in as-VHP T15 HSS were measured and reported in Table 2.

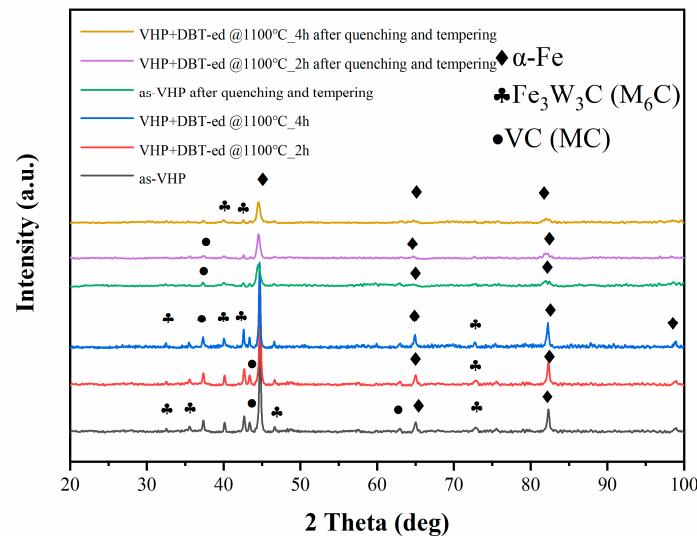


Figure 2. XRD patterns of T15 steel prepared by different processes.

Table 2. The volume fraction of carbide in T15 steel under different preparation processes.

Preparation Processes	Volume Fraction of Carbide(%)		
	M_6C	MC	$\text{M}_6\text{C}+\text{MC}$
as-VHP	9.1	17.0	26.1
as-VHP after quenching and tempering	6.2	10.4	16.6
VHP+DBT-ed@1100°C_2h	10.8	18.9	29.7
VHP+DBT-ed@1100°C_2h after quenching and tempering	5.9	11.4	17.3
VHP+DBT-ed@1100°C_4h	10.7	17.6	28.3
VHP+DBT-ed@1100°C_4h after quenching and tempering	6.7	12.8	19.5

Figure 3c shows the SEM BSE microstructure of VHP T15 HSS after quenching and tempering. Figure 3d shows the size statistical distributions of M_6C , MC, and combined $\text{M}_6\text{C}+\text{MC}$ carbides, together with the corresponding fitted lognormal distribution curves, in the VHP T15 HSS after quenching and tempering, indicating $d = 0.63^{+0.12}_{-0.10}$, $0.42^{+0.06}_{-0.05}$ and $0.53^{+0.11}_{-0.09}$ μm for M_6C , MC, and $\text{M}_6\text{C}+\text{MC}$ carbides, respectively. As compared with carbide sizes in the as-VHP T15 HSS (Figure 3b), carbide growth is almost negligible. The average size of $\text{M}_6\text{C}+\text{MC}$ carbides is smaller than that (0.94 μm) in the HIP (1130 °C for 3 h) T15 HSS after quenching and tempering with the similar processing parameters (austenitizing at 1200 °C for 5 min and triple tempering at 540 °C for 1 h) [26]. The volume fractions of M_6C and MC carbides in VHP T15 HSS after quenching and tempering were measured and reported in Table 2. After heat treatment, the volume fraction of carbides is reduced, which is because most of the carbides are redissolved into the matrix during quenching. Although nanoscale secondary carbides precipitated during tempering [31–33], they cannot be seen under SEM with the magnification in the present work. The same heat treatment process parameters lead to similar carbide volume fraction in T15 HSS after heat treatment.

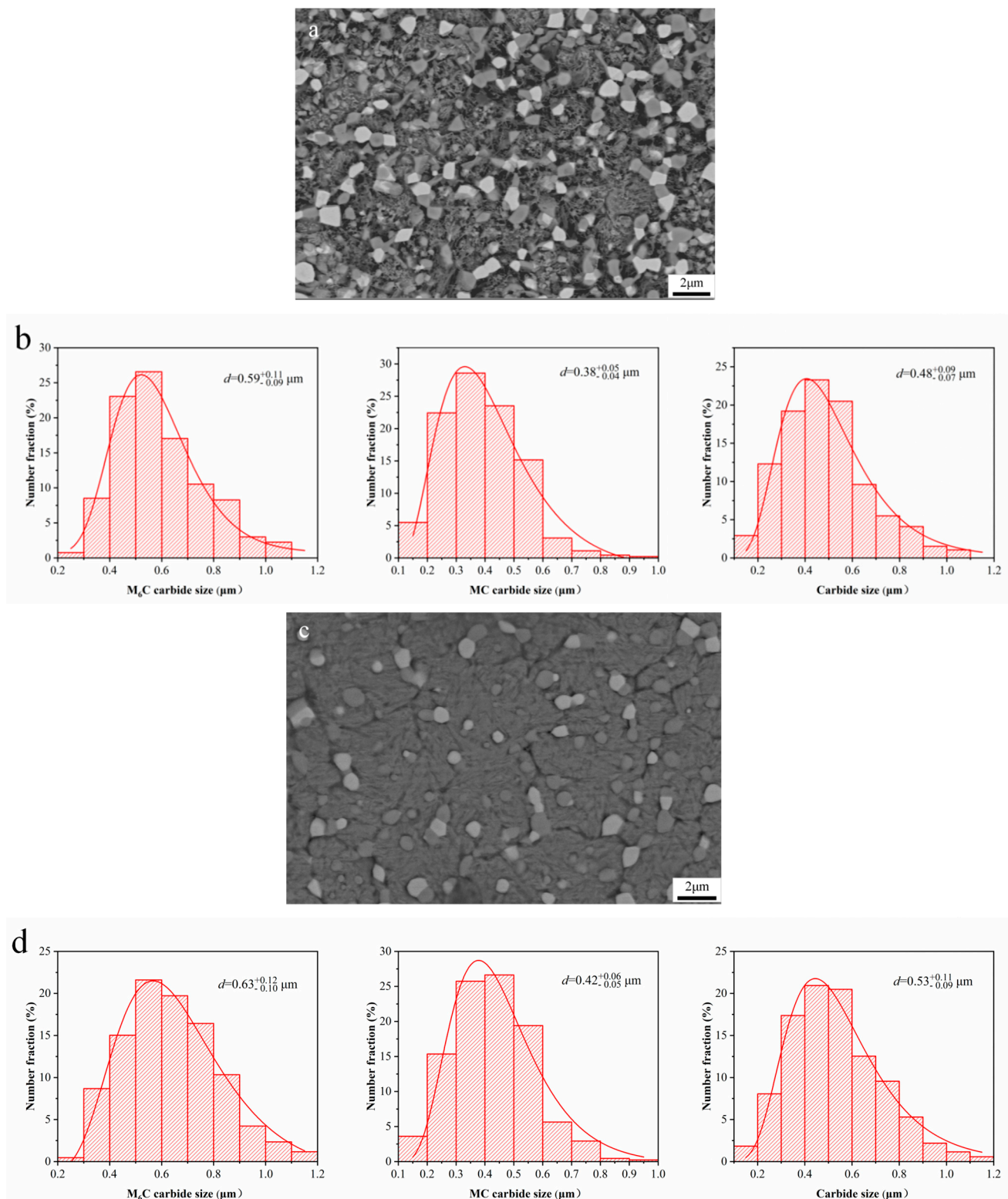


Figure 3. Microstructure diagram and carbide size distribution diagram of the as-VHP T15 HSS before and after quenching and tempering: (a) SEM BSE micrograph showing microstructure of the as-VHP T15 HSS; (b) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the as-VHP T15 HSS; (c) SEM BSE micrograph showing microstructure of the as-VHP T15 HSS after quenching and tempering; (d) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the as-VHP T15 HSS after quenching and tempering.

3.4. Microstructures in VHP+DBT-ed T15 HSSs

Figure 4a shows the SEM BSE microstructure of VHP + DBT-ed@1100°C_2h T15 HSS. Again, the microstructure is featured by uniformly distributed fine granular M₆C (brighter white) and MC (darker gray) carbides. The carbide size statistical distributions are shown in Figure 4b and the fitted lognormal curves reveal $d = 0.63^{+0.11}_{-0.10}$, $0.43^{+0.07}_{-0.06}$ and $0.54^{+0.11}_{-0.09}$ μm, respectively, indicating slight carbide growth during DBT relative to carbide sizes in the as-VHP T15. Figure 4c shows the SEM BSE microstructure of VHP + DBT-ed@1100°C_2h T15 HSS after quenching and tempering. Figure 4d illustrates the size statistical distributions of M₆C, MC, and M₆C+MC carbides, together with the corresponding fitted lognormal distribution curves, in the VHP + DBT-ed@1100°C_2h T15 HSS after quenching and tempering, indicating $d = 0.65^{+0.12}_{-0.10}$, $0.46^{+0.09}_{-0.07}$ and $0.55^{+0.11}_{-0.09}$ μm for M₆C, MC, and M₆C+MC carbides, respectively, similar to those in VHP T15 HSS after quenching and tempering. The volume fractions of M₆C and MC carbides in VHP + DBT-ed@1100°C_2h T15 HSS before and after quenching and tempering were measured and reported in Table 2.

Even when the DBT time was prolonged to 4 h, only slight carbide growth occurred during DBT of VHP T15 HSS at temperature of 1100 °C relative to carbide sizes in the as-VHP T15 HSS, as shown by the SEM BSE microstructure of VHP + DBT-ed@1100°C_4h T15 HSS in Figure 5a. This is confirmed by the carbide size statistical distributions as shown in Figure 5b, where carbide sizes are calculated by fitting the carbide size distributions with lognormal function: $d = 0.67^{+0.12}_{-0.11}$, $0.50^{+0.12}_{-0.10}$ and $0.58^{+0.14}_{-0.11}$ μm, still exhibiting slight carbide growth during DBT relative to carbide sizes in the as-VHP T15. Figure 5c shows the SEM BSE microstructure of VHP + DBT-ed@1100°C_4h T15 HSS after quenching and tempering. As shown in Figure 5d, after quenching and tempering, carbide sizes slightly increase, with $d = 0.68^{+0.15}_{-0.12}$, $0.53^{+0.10}_{-0.08}$ and $0.60^{+0.13}_{-0.11}$ μm, corresponding to M₆C, MC, and M₆C+MC carbides, respectively, also similar to those in VHP T15 HSS after quenching and tempering. The volume fractions of M₆C and MC carbides in VHP + DBT-ed@1100°C_4h T15 HSS before and after quenching and tempering were measured and reported in Table 2.

3.5. Density and Hardness

Figure 6 displays the relative densities of as-VHP and VHP + DBT-ed T15 HSSs, showing nearly full densification. The as-VHP T15 HSS exhibits nearly full densification with the relative density of $99.3 \pm 0.1\%$, as expected by the final position of the pressing punch. Of course, the VHP T15 still remains nearly full densification after DBT. The relative densities reached $99.5 \pm 0.2\%$ and $99.6 \pm 0.1\%$, respectively.

Figure 7 shows the Rockwell hardness histograms of VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSSs after quenching and tempering, exhibiting almost the same hardness values: 66.5 ± 0.4 , 66.7 ± 0.4 , and 66.4 ± 0.8 HRC, respectively. As compared with the HIP (1130 °C for 3 h) counterpart after quenching and tempering with the similar processing parameters (austenitizing at 1200 °C for 5 min and triple tempering at 540 °C for 1 h) [26], despite smaller primary carbides in VHP and VHP + DBT-ed T15 HSSs, they still exhibit hardness values similar to that in the HIP counterpart (67.5 HRC).

3.6. Three-Point Bend Fracture Strength

Figure 8 shows typical bend stress vs. bend displacement curves of quenched and tempered VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSSs. Based on testing of five specimens for each of the three T15 HSSs, bend fracture strength was obtained to be 3095 ± 99 , 3828 ± 121 , and 3620 ± 110 MPa, corresponding to VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSSs, respectively, showing much higher bend fracture strength of VHP + DBT-ed@1100°C_2h and VHP + DBT-ed@1100°C_4h T15 HSSs than that of the VHP T15 HSS. As compared with the HIP (1130 °C for 3h) counterpart after quenching and tempering with the similar processing parameters (austenitizing at 1200 °C for 5 min and triple tempering at 540 °C for 1 h) [26], despite smaller primary carbides in the VHP + DBT-ed T15 HSSs, the latter still exhibits bend fracture strength similar to that in the HIP counterpart (3700 MPa).

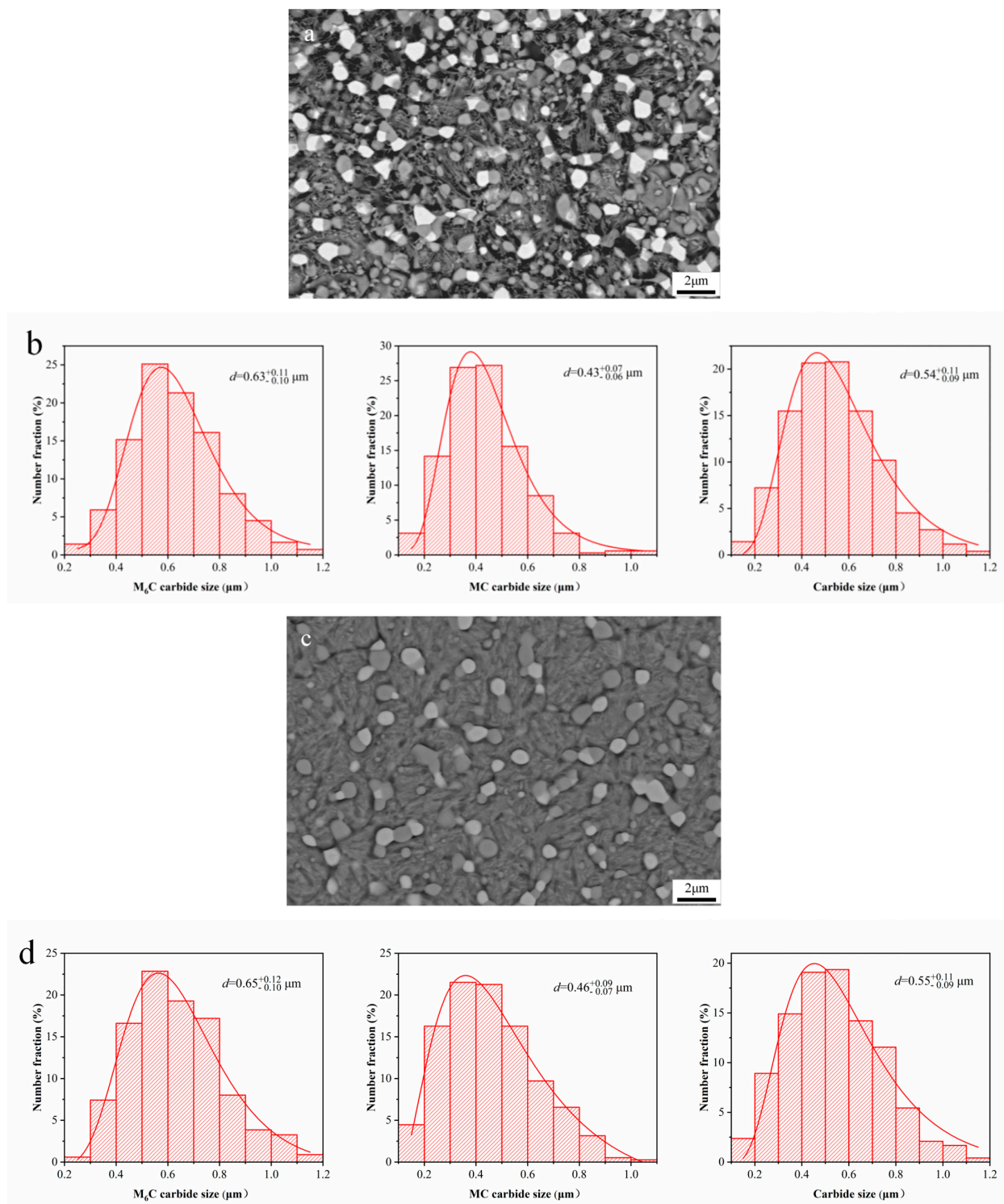


Figure 4. Microstructure diagram and carbide size distribution diagram of the VHP + DBT-ed@1100°C_2h T15 HSS before and after quenching and tempering: (a) SEM BSE micrograph showing microstructure of the VHP + DBT-ed@1100°C_2h T15 HSS; (b) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the VHP + DBT-ed@1100°C_2h T15 HSS; (c) SEM BSE micrograph showing microstructure of the VHP + DBT-ed@1100°C_2h T15 HSS after quenching and tempering; (d) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the VHP + DBT-ed@1100°C_2h T15 HSS after quenching and tempering.

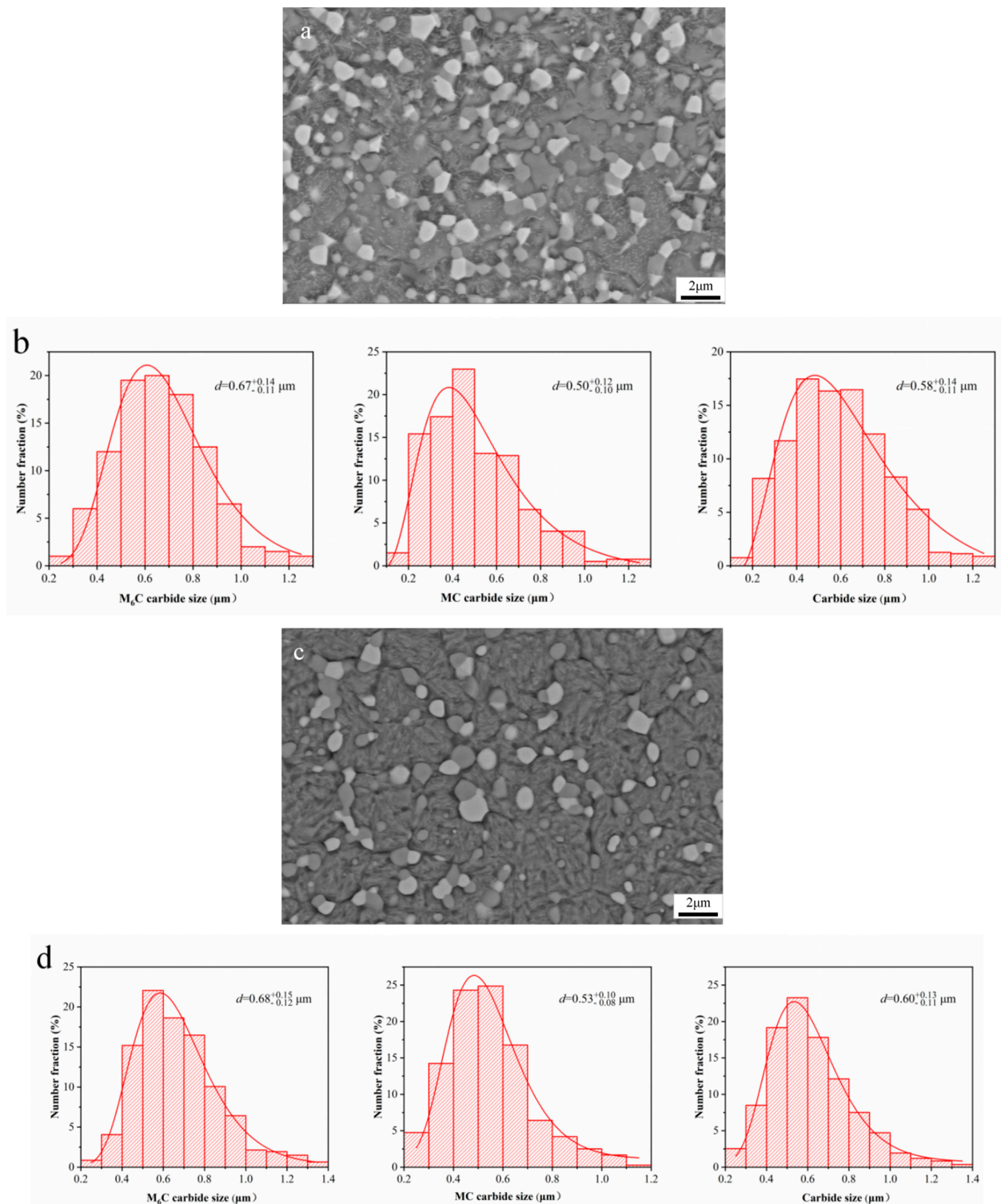


Figure 5. Microstructure diagram and carbide size distribution diagram of the VHP + DBT-ed@1100°C_4h T15 HSS before and after quenching and tempering: (a) SEM BSE micrograph showing microstructure of the VHP + DBT-ed@1100°C_4h T15 HSS; (b) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the VHP + DBT-ed@1100°C_4h T15 HSS; (c) SEM BSE micrograph showing microstructure of the VHP + DBT-ed@1100°C_4h T15 HSS after quenching and tempering; (d) size statistical distributions and fitted log-normal distribution curves of M₆C, MC, and M₆C+MC carbides in the VHP + DBT-ed@1100°C_4h T15 HSS after quenching and tempering.

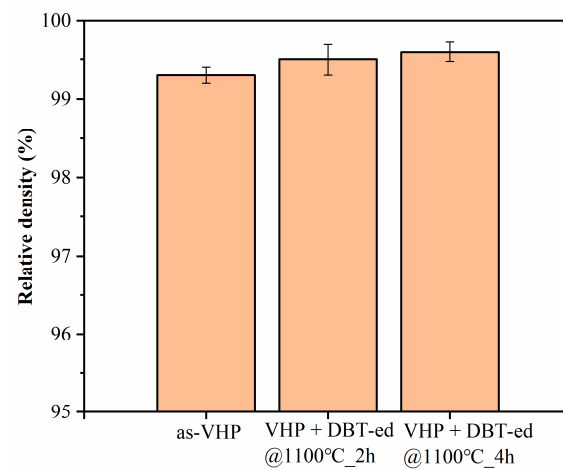


Figure 6. Relative density in the as-VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSS.

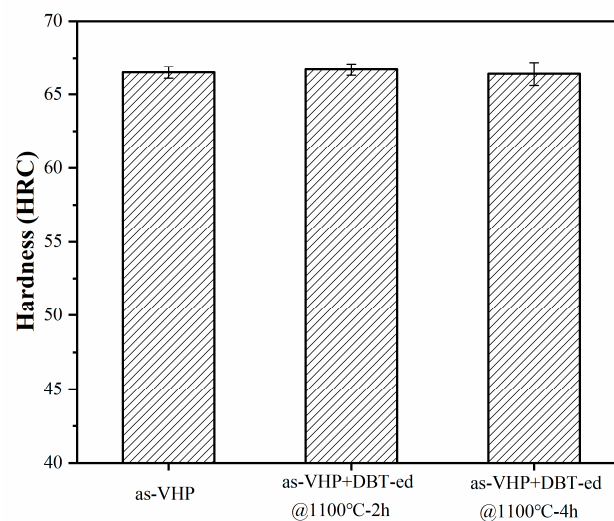


Figure 7. Rockwell hardness histograms of VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSSs after quenching and tempering.

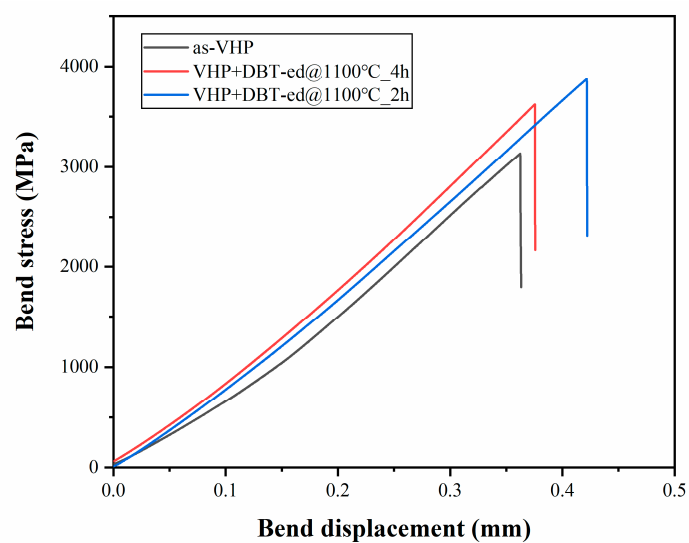


Figure 8. Bend stress vs. bend displacement curves of quenched and tempered VHP, VHP + DBT-ed@1100°C_2h, and VHP + DBT-ed@1100°C_4h T15 HSSs.

3.7. Fractured Surfaces of Three-Point Bend Specimens

Figure 9 shows the fractured surfaces of three-point bend specimens of VHP and VHP + DBT-ed T15 HSSs. In Figure 9a, most regions consist of protruded carbides and dents left by pulled-out carbides (comparable dimensions between carbides and dents), as marked by red and green arrows, respectively. This is a typical fractograph of quenched and tempered tool steels. Such fractograph reflects metallurgical bond between powders in these regions. However, in a few local regions rather than the aforementioned ones, the morphology comprising protruded carbides and dents is absent, and instead relatively flat morphology can be seen, as marked by dashed-line rectangles. By comparing with surface topograph of powders as shown in Figure 1c, these relatively flat regions should be considered as original powder surfaces, where metallurgical bond is not formed. In contrast, in Figure 9b,c, the almost entire fractured surfaces comprise protruded carbides and dents left by pulled-out carbides without the relatively flat morphology, suggesting significantly enhanced metallurgical bond between powders.

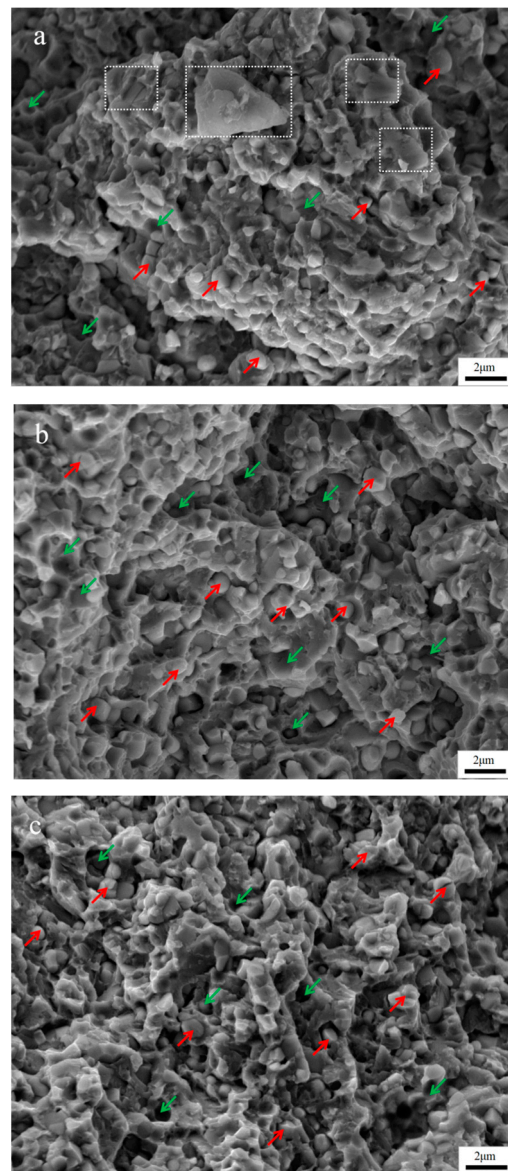


Figure 9. Fractured surfaces of three-point bend specimens of: (a) the as-VHP T15 HSS after quenching and tempering; (b) VHP + DBT-ed@1100°C_2h T15 HSS after quenching and tempering; and (c) VHP + DBT-ed@1100°C_4h T15 HSS after quenching and tempering.

4. Discussion

4.1. Microstructural Evolution during VHP and DBT

In the present work, T15 prealloyed powders were consolidated by VHP at a temperature as high as 1200 °C for a time as short as 15 min. Nearly full densification (relative density of $99.3 \pm 0.1\%$) was achieved. During powder consolidation by solid-state hot pressing in the present work, the primary mechanism for densification is the plastic and viscous flow driven by external stress [34,35]. Thus, the powder densifies rapidly under pressure and high temperature. The VHP temperature as high as 1200 °C did not cause excessive coarsening of carbides, and very fine carbides were still retained (carbide size distribution of $d = 0.48_{-0.09}^{+0.07} \mu\text{m}$), likely attributable to short time at the VHP temperature (15 min). This result can be rationalized by the well-known Ostwald ripening equation [36]: $\bar{d}^{-3} = \bar{d}_0^{-3} + K(D/RT)t$, where $\bar{d}_0$ is starting average carbide size, K is a solute concentration related constant, R is gas constant, T is temperature, $D = D_0 \exp(-E/RT)$ is solute diffusivity with D_0 being a constant and E being diffusion activation energy, and t is time. Despite high value of (D/RT) due to high T (1200 °C), short time t (15 min) still yields a low value of $K(D/RT)t$, leading to small average carbide size $\bar{d}$ after VHP. Negligible carbide growth during austenitizing (for quenching) at 1204 °C for 5 min can also be explained using similar arguments.

In the present work, T15 prealloyed powders were consolidated by VHP in solid state. Only slight shear deformation between powders occurred during VHP. Accordingly, mutual diffusion of elements at powder boundaries represents the primary mechanism underlying the formation of metallurgical bond between powders. The short time during VHP resulted in inadequate diffusion, leading to incomplete metallurgical bond between powders. This result is supported by the fractograph in Figure 9a, where non-metallurgical bonding regions are marked by dash-line rectangles as analyzed in Section 3.7.

During DBT at 1100 °C, long-time (2 and 4 h) mutual diffusion between the neighboring powder boundaries significantly enhanced metallurgical bond between powders. This result is supported by the fractograph in Figure 9b, which exhibits metallurgical bond in the entire regions as analyzed in Section 3.7. Despite prolonged DBT time (2 and 4 h), only slight carbide growth occurred as a result of relatively low DBT temperature (1100 °C). This result can also be rationalized by the aforementioned Ostwald ripening equation. Despite prolonged DBT time t (2 and 4 h), low values of (D/RT) due to low T (1100 °C) yield a low value of $K(D/RT)t$, leading to small average carbide size $\bar{d}$ after VHP.

4.2. Hardness of Consolidated T15 HSSs

The microstructure of a quenched and tempered HSS consists of micrometric to sub-micrometric primary carbides and nanoscale secondary carbides dispersed in the tempered martensite matrix [37,38]. The following factors critically affect HSS hardness: (1) solid solution strengthening of carbon and alloying elements to α -Fe lattice, (2) dispersion strengthening of primary and secondary carbides to the tempered martensitic matrix, and (3) the relative density or porosity [39]. During hardness testing, compressive load is applied. Thus, the hardness value is not sensitive to bonding quality between powders for PM HSSs. Based on the results presented above, the VHP, VHP+DBT-ed@1100°C_2h, and VHP+DBT-ed@1100°C_4h T15 HSSs after quenching and tempering have similar sizes (Figures 3d and 5d) and volume fractions (Table 2) of primary carbides. The same quenching and tempering process parameters generate similar carbon and alloying elements contents and similar secondary carbide volume fractions and sizes in the tempered martensite matrix. Based on the above discussion, solid-solution strengthening to α -Fe lattice and dispersion strengthening to the tempered martensitic matrix are similar in the three T15 HSSs after quenching and tempering. They have similar relative densities (Figure 6). The combination of these factors leads to similar hardness of the three T15 HSSs. Based on the above discussion, the difference in bonding quality between the VHP T15 HSS and

the VHP+DBT-ed T15 HSSs almost does not affect hardness. The HIP counterpart after quenching and tempering with the similar processing parameters [26] exhibits similar solid solution strengthening of carbon and alloying elements to α -Fe lattice and similar dispersion strengthening of secondary carbides to the tempered martensitic matrix. Due to primary carbides being one to two orders of magnitude larger than secondary carbides, as a result, the number of secondary carbides is much higher than that of primary carbides, dispersion strengthening of primary carbides is insignificant relative to that of secondary carbides. Thus, despite larger primary carbides in the HIP counterpart, the aforementioned three T15 HSSs in the present work still exhibit hardness values similar to that in the HIP counterpart.

4.3. Three-Point Bend Fracture Strength of Consolidated T15 HSSs

The bend fracture strength of a HSS is primarily determined by (1) strengthening effect, (2) carbide sizes, and (3) residual pores and non-metallurgical bond boundaries. The three T15 HSSs exhibit similar hardness and thus a similar strengthening effect. They also have similar carbide sizes and relative densities (residual porosity). Thus, the difference in bend fracture strength between VHP and VHP + DBT-ed T15 HSSs should originate from bond quality between powders. Indeed, as presented in Section 3.7, incomplete metallurgical bond between powders is present in VHP T15 HSS, whereas significantly enhanced metallurgical bond between powders is observed in VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs.

In addition, almost fully dense VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs, and fully dense HIP T15 HSS, have similar hardness and thus similar strengthening effect. Despite smaller carbides in VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs than those in the HIP T15, VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs exhibit three-point bend fracture strength similar to that in the HIP T15 HSS. This is likely attributable to lower extent of metallurgical bond in VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs than that in the HIP counterpart. Thus, the extent of metallurgical bond should be further improved in VHP+DBT-ed@1100°C_2h and VHP+DBT-ed@1100°C_4h T15 HSSs.

4.4. Proposals for Modification of Technological Parameters during VHP and DBT

The objective of the present work is to produce HSSs with large cross-sectional sizes by VHP using a VHP facility with low loading capacity. To do so, VHP is performed at high temperatures, in order to reduce pressure leading to full densification. In the present work, for T15 prealloyed powders, a VHP temperature of 1200 °C is utilized, which allows a pressure of 7 MPa. Further increasing VHP temperature to that approaching the solidus temperature (>1285 °C [40]) can minimize the pressure and thus maximize the cross-sectional sizes of produced T15. On the one hand, the VHP temperature approaching the solidus temperature tends to induce significant carbide growth. On the other hand, the minimized resistance of powders to the pressing punch movement increases the speed of the punch movement and thus minimizes the pressing time (e.g., <1 min), which limits carbide growth and maintains very fine carbides. Thus, VHP at the temperatures approaching the solidus temperature with minimized time (e.g., <1 min) is feasible, which maximizes the cross-sectional sizes of produced T15 while retaining very fine carbides. Similar to the case of VHP at 1200 °C for 15 min, complete metallurgical bond between powders cannot be attained in the case of VHP at temperatures approaching the solidus temperature for a very short time (e.g., <1 min). Hence, subsequent DBT is required. Based on the result presented above, DBT at 1100 °C for 4 h cannot achieve complete metallurgical bonding. Time of DBT at 1100 °C should be further prolonged for complete metallurgical bonding. Research work on VHP at temperatures approaching the solidus temperature with time of <1 min and DBT at 1100 °C for time of >4 h is on progress.

5. Conclusions

(1) In the present work, an innovative VHP approach for consolidation of highly alloyed HSS prealloyed powders was developed, which consists of high-temperature low-pressure short-time pressing and subsequent DBT. This VHP approach can be used to produce HSSs with large cross-sectional size by a VHP equipment having low loading capacity, and simultaneously to achieve full densification and complete metallurgical bond between powders while retaining fine carbides.

(2) T15 HSS prealloyed powders were consolidated using the new VHP approach. With the VHP temperature as high as 1200 °C, the pressure as low as 7 MPa leading to full densification was obtained, and almost full densification was attained for 15 min with pressure of 7 MPa and by monitoring the displacement of the pressing punch.

(3) As-VHP T15 HSS exhibits submicrometric carbide sizes smaller than those in the as-HIP counterpart but incomplete metallurgical bond between powders, due to the short processing time albeit the high VHP temperature. By subsequent DBT at a relatively low temperature of 1100 °C for 2–4 h, the extent of metallurgical bond between powders was significantly enhanced as a result of sufficient mutual diffusion at powder boundaries, while minimizing carbide growth due to the relatively low DBT temperature.

(4) After regular quenching and tempering, the VHP + DBT-ed T15 HSSs exhibit submicrometric primary carbide sizes smaller than those in the HIP counterpart after quenching and tempering with the similar processing parameters. Due to insignificant effect of primary carbide sizes on hardness, the VHP + DBT-ed T15 HSSs exhibit hardness similar to that in the HIP counterpart. Despite smaller primary carbide sizes in the VHP + DBT-ed T15 HSSs, they still exhibit three-point bend fracture strength similar to that in the HIP counterpart, likely attributable to a relatively lower extent of metallurgical bond between powders in the VHP + DBT-ed T15 HSSs.

(5) It is feasible to consolidate T15 prealloyed powders by VHP at a high temperature approaching the solidus temperature with very short timeframe (e.g., <1 min), which minimizes the pressure leading to full densification and thus maximizes the cross-sectional sizes of produced HSSs, while retaining very fine carbides. Full densification may be attained by DBT at 1100 °C for more than 4 h.

Author Contributions: Conceptualization, Y.L.; methodology, Y.L. and W.S.; software, W.S.; validation, W.S. and Y.L.; formal analysis, W.S.; investigation, W.S.; resources, Y.L.; data curation, W.S.; writing—original draft preparation, W.S.; writing—review and editing, Y.L.; visualization, Y.L.; supervision, Y.L.; project administration, Y.L.; funding acquisition, Y.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The raw and processed data required to reproduce these results are available by contacting the authors.

Acknowledgments: The present work was financially supported by the research funding from Wuhan University of Technology, China, for Newly Recruited Distinguished Professors (Grant number: 471-40120281).

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

References

1. Gsellmann, M.; Klünsner, T.; Mitterer, C.; Marsoner, S.; Leitner, H.; Boumpakis, A.; Skordaris, G.; Maier-Kiener, V.; Ressel, G. Influence of matrix composition and MC carbide content on damage behaviour of TiN-coated high speed steel due to cyclic shear and compression load. *Surf. Coat. Technol.* **2022**, *442*, 128546. [\[CrossRef\]](#)
2. Leitner, H.; Clemens, H.; Primig, S. On the evolution of secondary hardening carbides during continuous versus isothermal heat treatment of high speed steel HS 6-5-2. *Mater. Charact.* **2016**, *120*, 323–330.
3. Zhou, X.F.; Fang, F.; Jiang, J.Q.; Zhu, W.L.; Xu, H.X. Refining carbide dimensions in AISI M2 high speed steel by increasing solidification rates and spheroidising heat treatment. *Mater. Sci. Technol.* **2012**, *28*, 1499–1504. [\[CrossRef\]](#)
4. Liu, Z.Y.; Loh, N.H.; Khor, K.A.; Tor, S.B. Microstructure evolution during sintering of injection molded M2 high speed steel. *Mater. Sci. Eng. A* **2000**, *293*, 46–55. [\[CrossRef\]](#)

5. Xu, L.; Wei, S.; Xiao, F.; Zhou, H.; Zhang, G.; Li, J. Effects of carbides on abrasive wear properties and failure behaviours of high speed steels with different alloy element content. *Wear* **2017**, *376*, 968–974. [\[CrossRef\]](#)
6. Lu, L.; Hou, L.G.; Zhang, J.X.; Wang, H.B.; Cui, H.; Huang, J.F.; Zhang, Y.A.; Zhang, J.S. Improved the microstructures and properties of M3: 2 high-speed steel by spray forming and niobium alloying. *Mater. Charact.* **2016**, *117*, 1–8. [\[CrossRef\]](#)
7. Xu, H.; Shen, Y.; Cao, R.; Li, W.; Pang, Y.; Che, H.; Wang, T.; Liang, C.; Liu, S.; Qin, W.; et al. Effect of Different Rolling Passes on Microstructure and Mechanical Properties of M390 Powder Metallurgy High-Speed Steel. *J. Mater. Eng. Perform.* **2022**, *31*, 9650–9659. [\[CrossRef\]](#)
8. Yu, X.F.; Zheng, D.Y.; Yang, X.F.; Wang, S.Y.; An, M.; Yan, G.B.; Xia, Y.Z.; Xing, F. Effect of carbide precipitation behavior at high temperatures on microstructure and mechanical properties of M50 steel. *J. Mater. Res. Technol.* **2022**, *18*, 1155–1165. [\[CrossRef\]](#)
9. Li, T.; Zhong, Y.; Qu, S.; Zhang, Z. Influences of the characteristics of carbide particles on the rolling contact fatigue life of rare earth modified, highly clean bearing steel. *Eng. Fail. Anal.* **2023**, *143*, 106888. [\[CrossRef\]](#)
10. Gimenez, S.; Zubizarreta, C.; Trabadelo, V.; Iturriza, I. Sintering behaviour and microstructure development of T42 powder metallurgy high speed steel under different processing conditions. *Mater. Sci. Eng. A* **2008**, *480*, 130–137. [\[CrossRef\]](#)
11. Roberts, G.; Krauss, G.; Kennedy, R. *Tool Steels*, 5th ed.; ASM International: Materials Park, OH, USA, 1998.
12. Boccalini, M.; Goldenstein, H. Solidification of high speed steels. *Int. Mater. Rev.* **2001**, *46*, 92–115. [\[CrossRef\]](#)
13. Chen, P.; Liu, Y.; Ping, X.; Li, W.; Yi, Y. Influence mechanism of Y addition on microstructure, adsorbability, mechanical properties of as-cast high speed steel. *Mater. Sci. Eng. A* **2023**, *863*, 144520. [\[CrossRef\]](#)
14. Wießner, M.; Leisch, M.; Emminger, H.; Kulmburg, A. Phase transformation study of a high speed steel powder by high temperature X-ray diffraction. *Mater. Charact.* **2008**, *59*, 937–943. [\[CrossRef\]](#)
15. Trabadelo, V.; Giménez, S.; Iturriza, I. Development of powder metallurgy T42 high speed steel for structural applications. *J. Mater. Process. Technol.* **2008**, *202*, 521–527. [\[CrossRef\]](#)
16. Kumar, K.S.; Lawley, A.; Koczak, M.J. Powder metallurgy T15 tool steel: Part I. Characterization of powder and hot isostatically pressed material. *Met. Mater. Trans. A* **1991**, *22*, 2733–2745. [\[CrossRef\]](#)
17. Farayibi, P.; Blüm, M.; Weber, S. Densification of a high chromium cold work tool steel powder in different atmospheres by SLPs: Microstructure, heat treatment and micromechanical properties. *Mater. Sci. Eng. A* **2020**, *777*, 139053. [\[CrossRef\]](#)
18. Lin, Y.J.; Chen, J.R.; Zhang, L.Y. Microstructure of high carbon and vanadium die steel CPM10V after vacuum sintering. *Powder Metall. Technol.* **1997**, *15*, 255–257.
19. Wang, C.L.; Zheng, Q.; Jiang, W.B.; Li, H.X. Investigation on hot pressing of PM high speed steels at low pressure. *Powder Metall. Ind.* **2005**, *15*, 1–6.
20. Pellizzari, M.; Fedrizzi, A.; Zadra, M. Influence of processing parameters and particle size on the properties of hot work and high speed tool steels by spark plasma sintering. *Mater. Des.* **2011**, *32*, 1796–1805. [\[CrossRef\]](#)
21. Oh, S.-J.; Jun, J.-H.; Lee, M.-H.; Shon, I.-J.; Lee, S.-J. Microstructure and mechanical properties of highly alloyed FeCrMoVC steel fabricated by spark plasma sintering. *Met. Mater. Int.* **2018**, *24*, 597–603. [\[CrossRef\]](#)
22. Jin, J.; Gao, R.; Peng, H.; Guo, H.; Gong, S.; Chen, B. Rapid solidification microstructure and carbide precipitation behavior in electron beam melted high-speed steel. *Met. Mater. Trans. A* **2020**, *51*, 2411–2429. [\[CrossRef\]](#)
23. Li, Y.; Wang, Y.; Niu, J.; Liu, S.; Lin, Y.; Liu, N.; Ma, J.; Zhang, Z.; Wang, J. Microstructure and mechanical properties of M2 high speed steel produced by electron beam melting. *Mater. Sci. Eng. A* **2023**, *862*, 144327. [\[CrossRef\]](#)
24. Sinico, M.; Metelkova, J.; Dalemans, T.; Thijs, L.; Van Hooreweder, B. High speed laser powder bed fusion of M789 tool steel with an optimized 120 µm layer thickness approach. *Procedia CIRP* **2022**, *111*, 162–165. [\[CrossRef\]](#)
25. Lim, S.-H.; Ryou, K.; Jang, K.; Choi, W.S.; Lee, H.M.; Choi, P.-P. Hot cracking behavior of additively manufactured D2 steel. *Mater. Charact.* **2021**, *178*, 111217. [\[CrossRef\]](#)
26. Zhong, H.L.; Fang, Y.; Kuang, C.; Kuang, X.; Hao, Q.; Li, X. Development of powder metallurgy high speed steel. *Mater. Sci. Forum* **2010**, *638*, 1854–1859. [\[CrossRef\]](#)
27. Peng, H.; Hu, L.; Li, L.; Zhang, L.; Zhang, X. Evolution of the microstructure and mechanical properties of powder metallurgical high-speed steel S390 after heat treatment. *J. Alloys Compd.* **2018**, *740*, 766–773. [\[CrossRef\]](#)
28. Qiu, Y.; Lin, Y.J.; Zhang, Q.Y.; Chen, F.; Zhang, Y.X. Study on tool steels with ultrahigh carbon and ultrahigh chromium prepared by vacuum hot-pressing. *Powder Metall. Technol.* **2021**, *39*, 297–303.
29. Zepón, G.; Ellendt, N.; Uhlenwinkel, V.; Bolfarini, C. Solidification sequence of spray-formed steels. *Met. Mater. Trans. A* **2015**, *47*, 842–851. [\[CrossRef\]](#)
30. Crucible Industries Data Sheets. Available online: <https://www.crucible.com/Products.aspx?c=DoList> (accessed on 17 March 2023).
31. Wang, Y.; Chu, S.; Mao, B.; Xing, H.; Zhang, J.; Sun, B. Microstructure, residual stress, and mechanical property evolution of a spray-formed vanadium-modified high-speed steel processed by post-heat treatment. *J. Mater. Res. Technol.* **2022**, *18*, 1521–1533. [\[CrossRef\]](#)
32. Jovičević-Klug, P.; Puš, G.; Jovičević-Klug, M.; Žužek, B.; Podgornik, B. Influence of heat treatment parameters on effectiveness of deep cryogenic treatment on properties of high-speed steels. *Mater. Sci. Eng. A* **2022**, *829*, 142157. [\[CrossRef\]](#)
33. Čiger, R.; Krbaťa, M.; Kianicová, M.; Eckert, M.; Chochlíková, H. Effect of heat treatment on notch toughness of powdered tool steels M390 and M398. *Procedia Struct. Integr.* **2023**, *43*, 312–317. [\[CrossRef\]](#)
34. Blendell, J.E.; Rheinheimer, W. Solid-State Sintering. *Encycl. Mater. Tech. Ceram. Glasses* **2020**, *1*, 249–257. [\[CrossRef\]](#)

35. Saini, N.; Pandey, C.; Mahapatra, M.M.; Mulik, R.S. Evolution of nano-size precipitates during tempering of 9Cr-1Mo-1W-V-Nb steel and their influence on mechanical properties. *Mater. Sci. Eng. A* **2018**, *711*, 37–43. [[CrossRef](#)]
36. Porter, D.A.; Easterling, K.E. *Phase Transformations in Metals and Alloys*, 3rd ed.; Taylor & Francis: Milton Park, UK, 1992.
37. Hoyle, G. *High Speed Steels*; Butterworth: Cambridge, UK, 1988.
38. Xu, G.; Huang, P.; Wei, Z.; Feng, Z.; Zu, G. Microstructural variations and mechanical properties of deep cryogenic treated AISI M35 high-speed steel tempered at various temperatures. *J. Mater. Res. Technol.* **2022**, *17*, 3371–3383. [[CrossRef](#)]
39. Stiller, K.; Svensson, L.E.; Howell, P.R.; Rong, W.; Andren, H.O.; Dunlop, G.L. High resolution microanalytical study of precipitation in a powder metallurgical high speed steel. *Acta Metall.* **1984**, *32*, 1457–1467. [[CrossRef](#)]
40. Available online: <https://www.crucible.com/PDFs/DataSheets2010/dsT15v1%202010.pdf> (accessed on 17 March 2023).

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.