



Article

Minimizing Porosity in 17-4 PH Stainless Steel Compacts in a Modified Powder Metallurgical Process

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Abstract: Nowadays, powder-based manufacturing processes are recognized as cost-efficient methods frequently employed for producing parts with intricate shapes and tight tolerances in large quantities. However, like any manufacturing method, powder-based technologies also have several disadvantages. One of the most significant issues lies in the degree of porosity. By modifying the morphology of the gas-atomized spherical 17-4PH stainless steel powder via prior ball milling and then raising both the pressure of cold compaction (1.6 GPa) and sintering temperature (1275 °C), the porosity could be reduced considerably. In our novel powder metallurgical (PM) experimental process, an exceptionally high green density of 92% could be reached by employing die wall lubrication instead of internal lubrication and utilizing induction heating for rapid sintering. After sintering (at temperatures of 1200, 1250, and 1275 °C), the samples aged in the H900 condition were then mechanically tested (Charpy impact, HV hardness, and tensile tests) as a function of porosity. Sintering at 1275 °C for one hour enabled porosity reduction to below 4%, resulting in 1200 MPa yield strength and 1350 MPa ultimate tensile strength with significant (16%) fracture strain. These values are comparable to those of the same alloy products fabricated via ingot metallurgy (IM) or additive manufacturing (AM).

Keywords: 17-4PH stainless steel; powder metallurgy; porosity; mechanical properties; corrosion resistance; fracture mechanism



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1. Introduction

Powder metallurgy (PM) is a cost-effective way to produce near-net-shape metallic parts for the automotive, aeronautical, medical, and other engineering industries. In the world of PM, the conventional press and sinter (P&S) method is the most used one to produce iron-based products for mass production [1]. This technology is primarily employed for low-alloyed steel powders, particularly to manufacture parts with complex geometry where outstanding strength is not the main priority. PM is currently even less applicable to highly alloyed stainless steels due to production difficulties.

Compared to commercial Fe-based alloys, 17-4PH stainless steel (SS) does not just present excellent corrosion resistance like the 304 grade SS but also outstanding strength due to the copper-based precipitates formed during the aging heat treatment. By selecting the appropriate aging treatments, its strength and toughness can be adjusted within wide limits according to the requirements of the manufactured parts. This alloy can be considered as the workhorse of SS additive manufacturing. Its high resistance for compression, however, makes it difficult to be compacted to the desired shape from powder by conventional cold compaction. In general, this type of alloy in powder form is rather used in metal injection molding (MIM) or in the field of additive manufacturing (AM), e.g., selective laser melting

(SLM) and metal binder jetting (MBJ) [2–4]. In these cases, the powder particles do not need to be plastically deformed and cold welded to each other during the shaping process. Table 1 shows typical porosity values and the related mechanical properties in the case of 17-4PH alloy made by using different technologies. Obviously, wrought products can be used as a reference when comparing different properties because they are considered non-porous materials. Based on the values shown in Table 1, it can be concluded that, except for P&S technology, all of the rest of the powder-based technologies can result in similar values as in the case of wrought steel. Of course, these values also significantly depend on the heat treatment condition.

Table 1. Typical porosity content and mechanical properties of 17-4PH SS manufactured by different technologies.

Applied Technology	State	Porosity (%)	Hardness	Yield Strength (MPa)	Tensile Strength (MPa)	Fracture Strain (%)	Impact Energy (Joule)	Ref.
IM (wrought)	H900	0	43 HRC				14.68	[5]
	H900	0	39 HRC	1170	1390	5		[6]
	H900	0	45 HRC	1379	1448	7		[7]
	H900	0	>38 HRC	>1070	>1170	>10	>6.8	[8]
MIM	1300 °C for 4 h	2.3	280 HV	765	770	2		[9]
	1350 °C for 1 h	<1			1000	7		[10]
	H900	<3	437 ± 15 HV	1113 ± 27	1346 ± 16	7.8 ± 1		[11]
3D Printing	H900	<1	42 HRC	1200	1368	2.6		[6]
		<1	31 ± 1 HRC		1090 ± 30	22 ± 0.5		[12]
		<2.5	445 HV	1190 ± 16	1309 ± 13	3.7 ± 0.7	6	[3]
MBJ	H900	2.65		1079 ± 33	1294 ± 9	8.3 ± 3.6		[4]
	H900 + HIP	1.18		1228 ± 74	1407 ± 15	15.5 ± 3		[4]
P&S	690 MPa; 1340/1 h H900	14.2	355 HV10		1147	2.4		[13]
	760 MPa; 1275/45 min	7	448 HV100	1048	1136	1.4		[14]
	H900	7			1080	4.2		[15]

There are only a few studies in the literature that deal with producing parts from 17-4PH SS powder with traditional PM technology [1,14–16]. The main reason for this is that this alloy is difficult to cold press. While the porosity content of the cold compacted pure copper pressed at 600 MPa is around 7% [17], in the case of 17-4PH, this value is around 23% [16]. During compaction, the size, morphology, and contamination (oxygen content) of the powder surface play a significant role. Generally, these properties are determined by the selected and applied powder fabrication route. There are many different methods for producing powders, but among them, at the industrial level, gas- and water-atomization are the most important. Gas-atomization ensures rather spherical morphology and higher purity, while water-atomization causes rather irregular morphology and higher contamination. The spherical morphology ensures good flowability and high apparent density for the powder, the properties of which are favorable for MIM technology and AM methods. Samples made by SLM from gas-atomized spherical 17-4PH powder show lower porosity and higher mechanical properties than the samples made from water-atomized powder [12]. However, this type of spherical powder with high apparent density is not optimal for the cold pressing process. Irregular powder with lower apparent density results in greater compaction and cold-welding of particles on pressing, resulting in a stronger green body with higher green strength. Obviously, the surface area between the compacted powder particles in a green sample has a key effect on the whole process. The latter can be increased using high compacting pressure to minimize the distances and the volume of porosity [17]. If neighboring particles are closer together, less atomic movement is required

during sintering. Based on Raynova et al., the green density of the powder compact has the greatest effect on both the strength and ductility of the sintered samples [18]. Moreover, the higher green density results in less density variation inside the sample and shape distortion after sintering [19]. The typical applied compaction value of the 17-4PH water atomized powder is between 400–800 MPa based on our previous research [1]. In the world of PM, water-atomized powder is generally utilized. It is cheaper than the gas-atomized powder, while the irregular shape and rough surface obtain stronger connections and interlockings between the adjacent powder particles. The disadvantage of this morphology is that high porosity remains between the irregular particles after cold pressing. To improve the yielding properties and decrease the friction, different types of process control agents and lubricants are used. These additives also help reduce the ejection force, but on the other hand, the internal lubricant decreases the green strength [20]. The porosity content of the green samples is relatively high (15–30%), and decreasing it below 10% is quite difficult [21], as most of the voids are filled with the applied lubricant.

At the beginning of sintering, the applied lubricant must be removed. This process is usually the first step in sintering and is called degassing. This process usually takes place at low temperatures and lasts several hours. Kazior et al. utilized thermal debinding at 450 °C to remove the residual binder from the cold compacted samples [13]. After that, sintering was conducted in dry hydrogen at 1340 °C for 1 h and reduced the porosity from 23% to 12%. The residual lubricant between the particles can contaminate the material with carbon, which is accompanied by deterioration of the properties [22]. Sintering should take place in a protective atmosphere, typically between 1200 and 1400 °C, for several hours to protect the material from oxidation and to minimize the value of porosity. Sintering is undoubtedly the most expensive step in the entire process. For this purpose, electric-heated or gas-fired furnaces are used in industry. However, the advantage of induction heating is that the heat is produced directly in the sample; therefore, the heat loss is lower. This heating method also enables the sintering time to be shortened [23]. Raynova et al. used this technique to sinter commercially pure titanium compacts. Only 15 min of sintering time was enough to bring the porosity of the samples below 2% [18]. The main objective during the sintering is to eliminate the gaps between the powder particles with atomic movements. During densification, the center of the neighboring particles should be closer to each other. The rate of this process mainly depends on the applied temperature and time of this kind of heat treatment. Sung et al. investigated the effect of sintering temperature on the density and shrinkage at the temperature range of 900 °C to 1350 °C. Based on this result, there is a linear function between the applied sintering temperature and density, or shrinkage [10]. Sintered density is strongly dependent on the initial green density and the utilized sintering parameters. Compared to the different powder-based technologies, the P&S samples show the highest porosity. Based on the few available sources in the literature, this value is between 6 and 12% [14], while in the case of SLM or MIM technology, this can be close to 1% [6,9].

The main goal of this work was to use powder with slightly changed morphology and size in P&S technology to successfully produce samples with significantly lower porosity than those produced before by others. The main modifications include changing the shape of 17-4PH gas atomized powder via ball milling, the lack of internal lubrication during cold pressing, the extremely high applied compaction force, and the utilization of induction heating during rapid one-hour sintering. Furthermore, another objective was to investigate the effect of sintering temperature on the porosity and the different mechanical properties of the prepared samples.

2. Materials and Methods

In this research, 17-4PH SS powder provided by Oerlikon was used for the P&S process. The chemical composition of the powder can be seen in Table 2.

Table 2. Chemical compositions of 17-4 PH SS powder (wt.%) based on the certificate of delivery.

Fe	Cr	Ni	Cu	Si	Mn	Nb	P	S	N	C
73.16	16.86	4.63	4.00	0.44	0.44	0.31	0.0019	0.004	0.03	0.02

The powder was produced by gas atomization, and it was primarily developed for 3D printing. The particle size range is between 15 and 45 μm . The powder consists mostly of spherical particles, as shown in Figure 1. However, almost all of the spherical particles contain satellites on the surfaces (Figure 1b). It can also be seen in Figure 1b that the surface of the spherical powder particles is made up of small grains with rough surfaces.

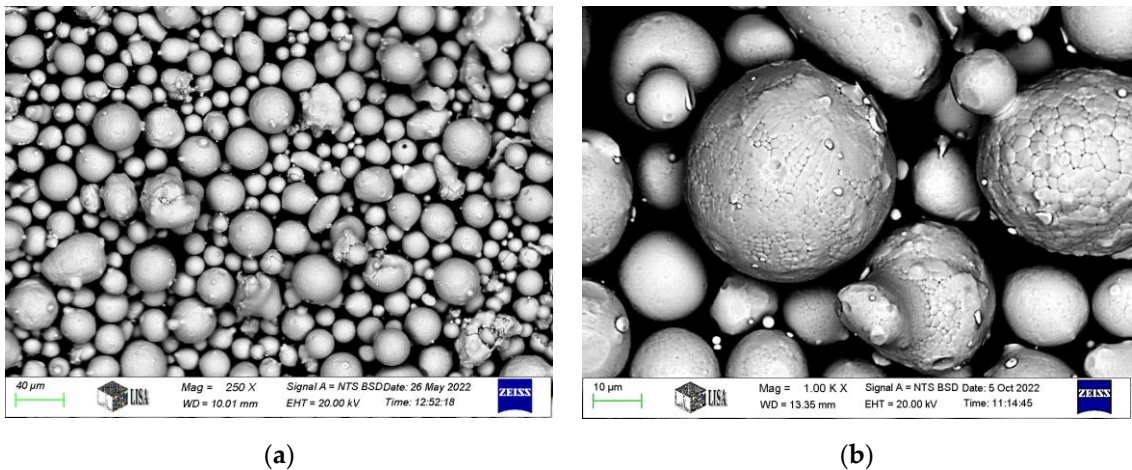


Figure 1. SEM micrographs of (a) gas-atomized spherical powder particles in low magnification (250 $\times$), (b) high magnification (1.00K $\times$) image showing satellite particles on the rough surface.

This powder morphology was not suitable to produce the 11 $\times$ 11 $\times$ 60 mm pieces required for the tests. Since only a weak bond was formed between the spherical particles during compaction, the green compacts disintegrated when ejecting from the die. That was the main reason for attempting to alter the morphology of the initial powder. For this purpose, planetary ball milling was utilized. The flow chart of the whole experimental work can be seen in Figure 2.

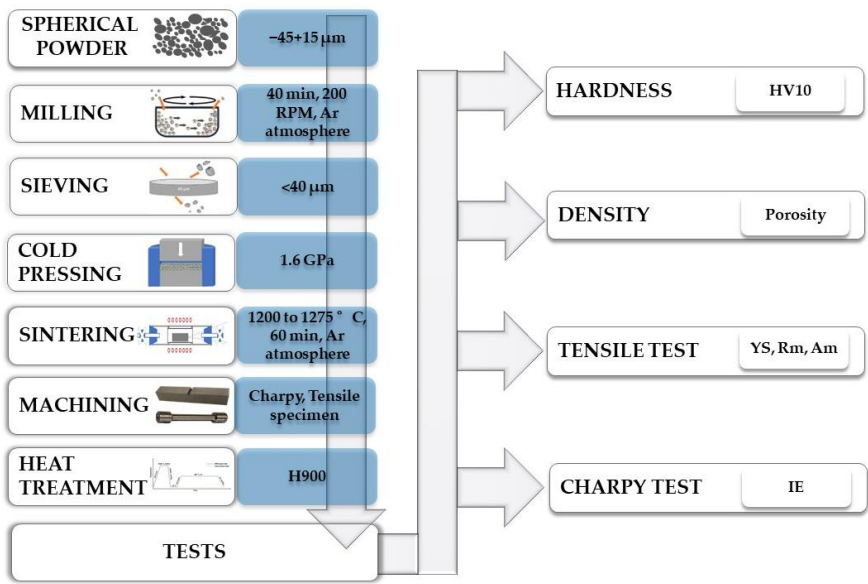


Figure 2. Flow chart of the utilized PM processing steps and testing methods.

The milling was carried out in a Fritsch Pulverisette 5 high-energy planetary ball mill (Fritsch, Idar-Oberstein, Germany) at room temperature for 40 min. The milling was performed in an Argon shield atmosphere (6.0 Siad, Miskolc, Hungary) using a 250 mL hardened steel vial and hardened steel balls with 8 mm diameter. The ball-to-powder weight ratio and rotational speed were 30:1 and 200 rpm, respectively. The applied close-to-optimal milling parameters were determined after performing many preliminary experiments. Before the cold pressing, the milled powder was sieved below 36 μm particle size with a Fritsch A3 IEC sieve shaker (Fritsch, Idar-Oberstein, Germany). A 1000 kN electro-hydraulic press was used for the consolidation of the powders to a rectangular 11 $\times$ 11 $\times$ 60 mm shape of green parts at ambient temperature at 1.6 GPa pressure in self-developed hardened steel die between hardened steel punches. This pressure value was maintained for 30 s before the ejection step. Graphite 33 spray (Kontakt Chemie, Iffezheim, Germany) was used as lubricant only on the die walls to decrease the friction and the ejection force. In order to prevent carbon contamination, internal lubrication and process control agents were not used during the cold pressing. Owing to this, degassing was not necessary to apply. The sintering of the green samples was carried out in a self-developed induction sintering machine in an Ar 6.0 protecting atmosphere. The sintering temperatures were 1200, 1250, and 1275 $^{\circ}\text{C}$ respectively. Five green samples were sintered at each temperature. The heating and cooling rates were 100 $^{\circ}\text{C}/\text{min}$, and the holding time was 1 h during the sintering. The sintered specimens were aged to H900 in an Argon atmosphere. The morphology of powders, green samples, and sintered samples was investigated by scanning electron microscopy (SEM) using a C. Zeiss EVO MA 10.

The density of the green samples was determined by the dimensional method. The Archimedes method was used to calculate the density of sintered samples with an Electronic Density Index Balance MK 2200 (MK-GMBH, Stahlhofen am Wiesensee, Germany). To calculate the porosity, 7.75 g/cm^3 theoretical density was applied. The hardness of the sintered samples was measured with the Vickers method (HV10) on a Wolpert UH930 (Illinois Tool Works, Shanghai, China). Tensile tests were carried out on the sintered samples in order to determine yield strength (YS), tensile strength (UTS), and the fracture tensile strain (ϵ_f). These tests were conducted with an Instron 5982-type universal material tester (Norwood, MA, USA) with a crosshead speed of 3 mm/min. Charpy tests were conducted according to ASTM E23-16b, using a Schenck-Trebel of 300 J capacity.

3. Results

3.1. Effect of Milling on the Powder

During planetary ball milling, continuous collisions take place between the bearing balls and powder particles. As a result, each powder particle in the milling jar undergoes some kind of plastic deformation and changes its shape. Figure 3a shows the particles after ball milling.

When the shape of the milled powder (Figure 3a) is compared to the SEM micrograph in Figure 1a, it is noticeable that the original spherical morphology has completely changed. Most of the ball-milled particles became sub-rounded and presented a metallic shiny surface. Based on the latter, it can be assumed that the inevitable surface oxide layer during atomization has partially or completely disappeared, although no chemical analysis was made in this regard. Some of the particles deformed more than the others and created flaky shapes (red arrows in Figure 3a). Moreover, these are, in most cases, cold welded to each other. This kind of shape is unfavorable in terms of green density because it prevents the rearrangement of the particles during the first stage of compaction and results in large voids and uneven properties. Since the width of these flakes is significantly larger than the other dimensions, they can be filtered out using a sieve. Before the cold pressing, the milled powder was sieved below 36 μm particle size, and thus, flaky particles of undesirable size and shape were removed. At the end of the process, powder with a smooth surface and a sub-rounded shape was obtained, which can be successfully compacted to the desired

geometry. During sieving, less than 20 wt.% of the milled powder was removed. Further ball-milling optimization is required in the future to reduce these losses.

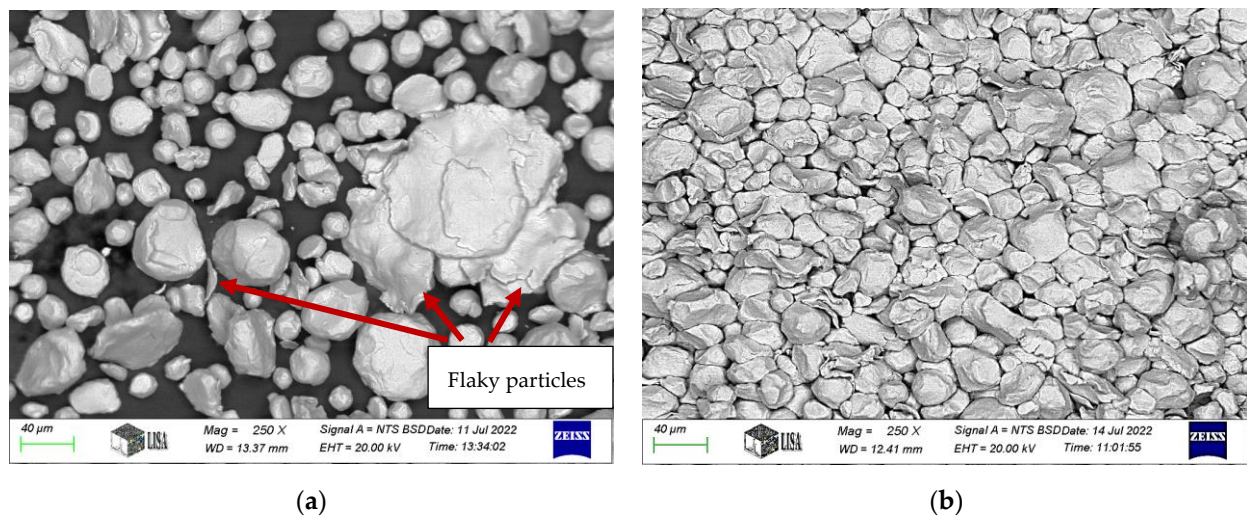


Figure 3. SEM micrographs: (a) milled powder particles after 40 min milling before sieving under 36 µm; the red arrows pointing at examples of flattened flaky like particles (b) a fractured surface of a cold-pressed green sample.

It is well known that the green strength seems to be directly proportional to the contact area between powder particles [17]. In Figure 3b, it can be clearly seen that due to the applied 1.6 GPa uniaxial pressure, the sub-rounded particles are close to each other and fill the space well inside the green compact. Due to the modified powder shape, the amount of cold-welded surface is larger compared to the case when the original spherical powder was used. The relative density of the green samples was between 91 and 92%, measured by the dimensional method. This green density is significantly higher than the published results so far, although there are no data for such a high (1.6 GPa) cold press value [1].

3.2. Densification during Sintering at Different Temperatures

Figure 4 shows the effect of applied sintering temperature on the densification process. After 1 h sintering at 1200 °C, most of the original boundary of cold-compacted powder particles can still be seen (Figure 4a). The dark lines represent the boundaries between adjacent particles right after sintering. The continuous line becomes dashed in some places, which indicates that the sintering process has already started between the neighbors, mostly where they were previously cold-welded together. This sintering temperature, however, is relatively low based on the literature [14–16]. The samples had a shrinkage of less than 4% during one hour of sintering.

When the applied temperature is increased by 50 °C up to 1250 °C, however, signs of sintering progress are already visible (Figure 4b). In this case, the original particle boundary is no longer clearly apparent. The dark triangles and black dots show that sintering did not completely take place; however, the porosity significantly decreased. Increasing the sintering temperature to 1275 °C, the black triangles, which represent the boundary of three particles, are no longer visible (Figure 4c). At this temperature, the separation of the alloying elements can also be observed. Based on the EDS measurements, in the dark gray areas, the Cr content is 21.9 wt.%, and the Ni content is 2.5 wt.% (Figure 4d). In contrast, in the light gray areas, the Cr content is 16.2 wt.%, and the Ni content is 5.3 wt.%. The applied temperature, therefore, affects not only the degree of porosity but also the rearrangement of the alloying elements. It is also important to note that a significant amount of Mn and Si-based spherical inclusions can be found in the microstructure (purple arrows in Figure 4d). They were created during powder atomization and have negative effects on the properties of the material. Figure 5 shows the calculated porosity after sintering at different

temperatures. These porosity values determined from the density measurement confirm the differences that can be seen in the previous SEM images. When the applied sintering temperature increased from 1200 °C to 1275 °C, the porosity decreased from 6.9% to 3.8%. Samal et al. utilized a 760 MPa cold press at the same 1275 °C temperature and obtained a significantly higher (7%) porosity after sintering. This also highlights the importance of the applied cold-press value on the efficiency of sintering.

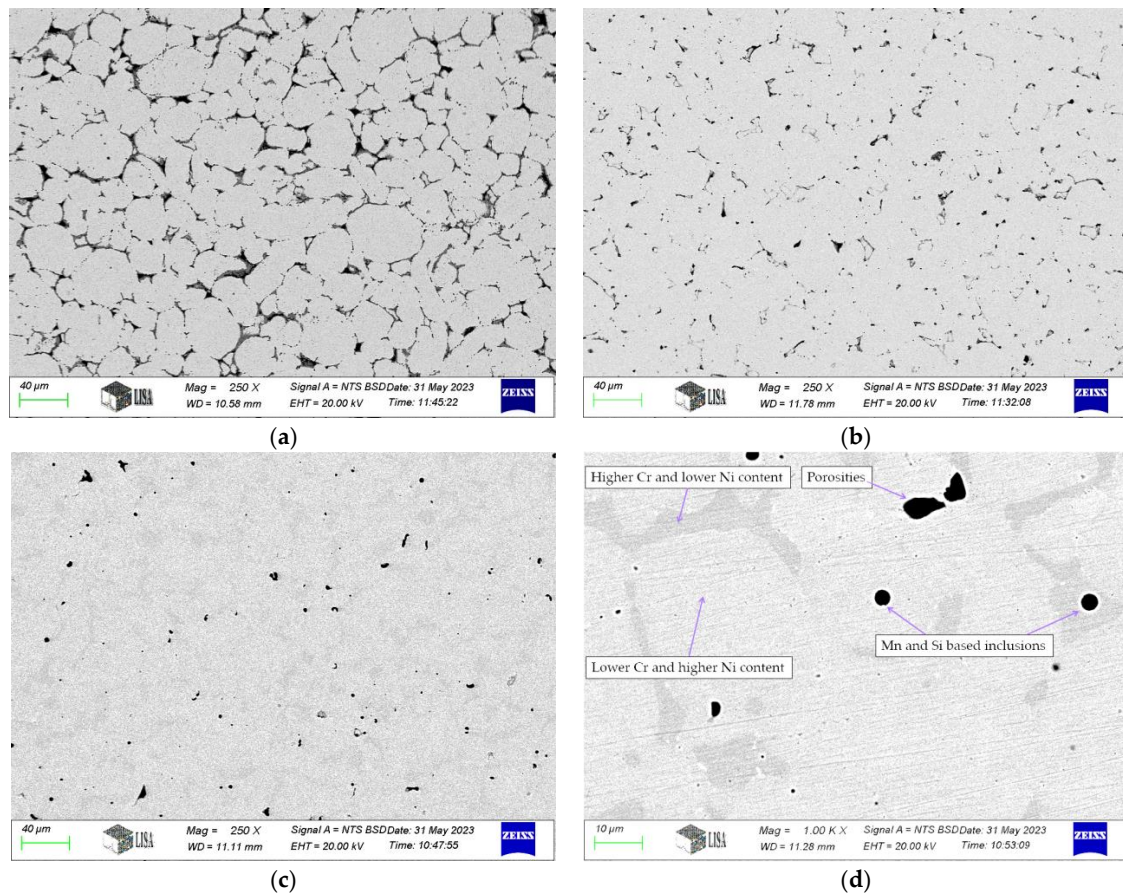


Figure 4. SEM micrographs: (a) sintered at 1200 °C for 60 min; (b) sintered at 1250 °C for 60 min; (c) sintered at 1275 °C for 60 min; (d) sintered at 1275 °C for 60 min.

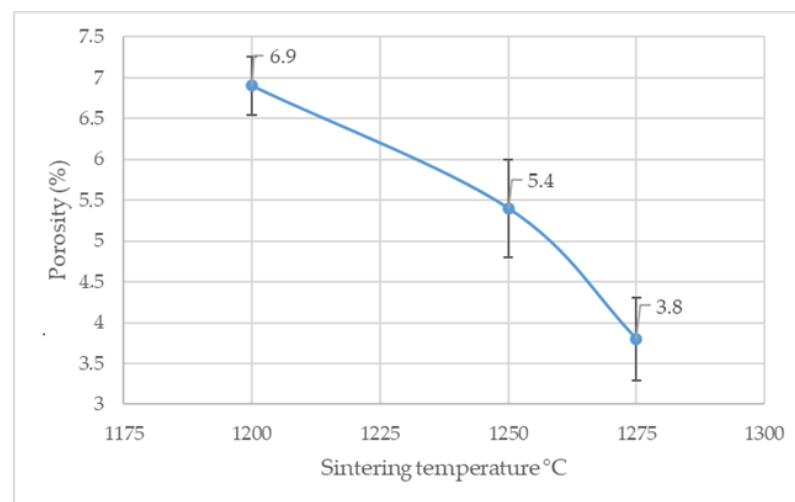


Figure 5. Calculated porosity values of 17-4PH SS (H900) samples after sintering at different temperatures for 1 h.

3.3. Hardness

The hardness of an alloy depends on its composition, microstructure (grain size, type, and quantity of phases), and porosity. The latter is particularly important when it comes to materials made from powder. Based on the utilized measurements, porosity has a significant effect on hardness. This is because porosity under a tested surface spot, under a given load, will have a deeper indentation than solid material. This leads to a lower hardness value. The measured hardness values of the sintered and aged (H900) samples are depicted in Figure 6.

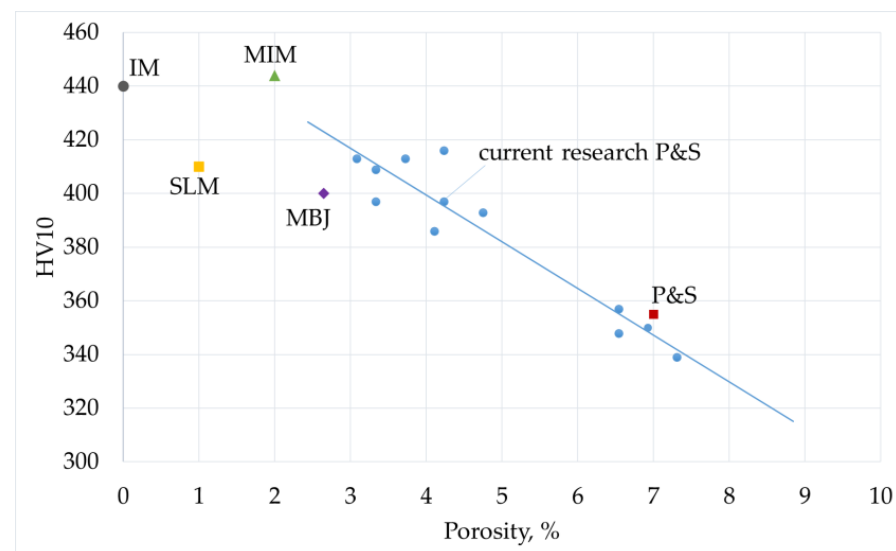


Figure 6. Hardness vs. porosity values of 17-4PH SS (H900) samples produced by different techniques (IM [7], SLM [6], MIM [11], MBJ [4], and P&S [13]).

The test results show a linear correlation with the porosity content. Reducing the porosity from 7.4% to 3.2% results in an increase in hardness from 339 HV to 412 HV. Figure 6 also includes some literature data [4,6,7,11,13] for samples produced using different methods. Overall, it can be concluded that the values measured during this research match those in the available literature. Of course, the different technologies also affect the grain structure and, thus also, the hardness. This is why the hardness from similar (P&S) [13] technology fits better with the measurement trend.

3.4. Tensile Properties

Tensile testing is a very useful method that can be used to test not only the strength of the material but also its deformability. It is important to find the optimal balance between these properties because brittle material is not suitable for engineering applications, even if its strength is extremely high. In the case of powder metallurgy products, a deformation capacity of at least 6% is mandatory. Since the porosity can significantly reduce the strain properties of the material, it is very important to decrease it. Figure 7 shows how the tested samples respond to tensile stress in the case of different sintering temperatures.

During the tensile tests, all the prepared P&S samples showed more than 6% plastic strain before the break. The highest fracture strain (12.2%) corresponds to the highest applied sintering temperature (1275 °C). Figure 7 also contains some already published tensile curves of the same alloy, made with a different technology. The determined strength values (YS, UTS) as a function of porosity are shown in Figure 8.

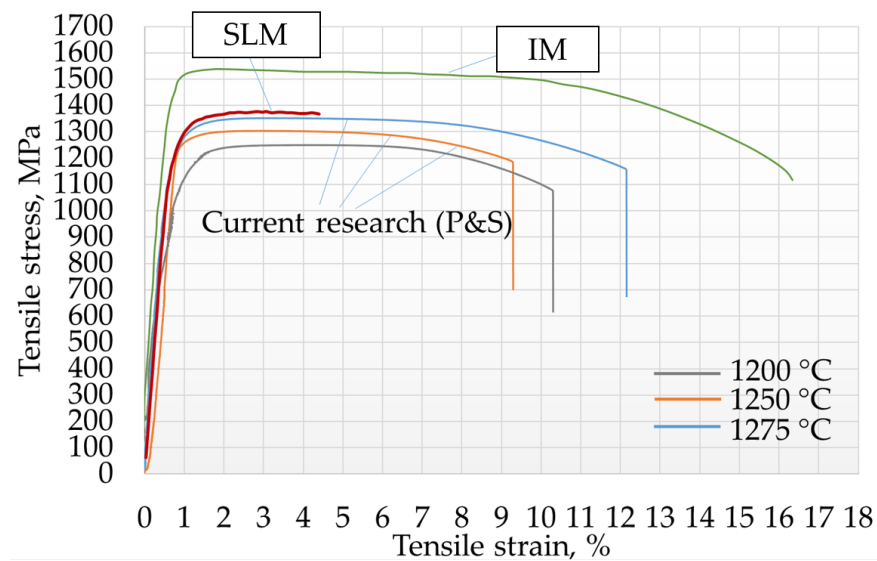


Figure 7. Tensile curves of 17-4PH SS (H900) samples produced by different techniques (IM [24], SLM [23], and P&S).

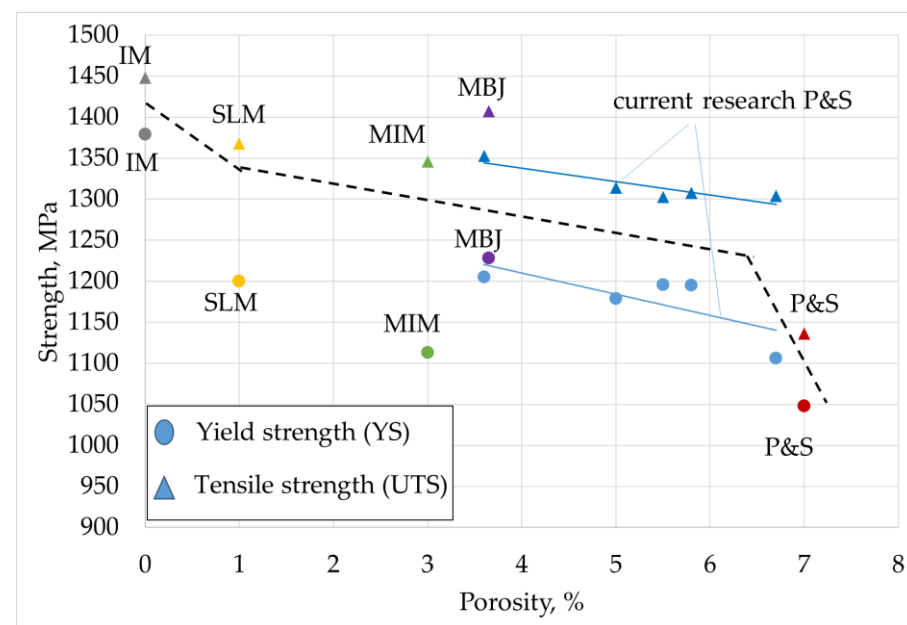


Figure 8. Strength vs. porosity values of 17-4PH SS samples produced by different techniques (IM [7], SLM [6], MIM [11], MBJ [4], and P&S [14]).

Both the yield strength and the tensile strength increase linearly as the porosity decreases. Some literature data can also be seen in Figure 8 to compare the results of the applied novel technology with the results of other existing methods. Obviously, porosity-free wrought materials produced using conventional ingot metallurgy have the highest strength values, although our own results are close to these (Figures 7 and 8).

3.5. Charpy Test

From the point of view of the practical usability of the materials, an important question is how the material behaves in the case of impact-like stress. The toughness of an alloy depends primarily on hardness and internal defects such as porosity. The toughness of porosity-free 17-4PH SS is mainly dependent on the heat-treated condition. Table 3 shows the hardness (HRC) and impact energy (KV23) values in the case of different aged states.

Based on the data in Table 3, it can be concluded that hardness and impact energy change in opposite directions.

Table 3. Hardness and impact energy values of the porosity-free 17-4PH SS produced via IM at different heat-treated conditions.

	H900	H925	H1025	H1075	H1100	H1150	H1150M
HRC	>40	>38	>35	>32	>31	>28	>24
KV23 (J)	n.d.	>6.8	>20	>27	>34	>41	>75

Hideki et al. showed that the toughness of 17-4PH SS produced by MIM technology is lower than their wrought counterparts [24]. Porosity acts as a stress concentrator and is considered the starting point of cracks. Suri et al. compared the impact energy of sintered and wrought 17-4PH samples [5]. Notched and unnotched specimens were used at different heat-treated conditions. Results showed that the notched wrought sample in an H900 state demonstrated around 14 Joules of impact energy, while the isostatic cold-pressed and sintered samples with 1% porosity demonstrated 2 J impact energy. The 17-4 PH SS is a notch-sensitive material, which is reflected in the impact properties of the sintered and wrought material. Figure 9 shows the relationship between hardness and impact energy.

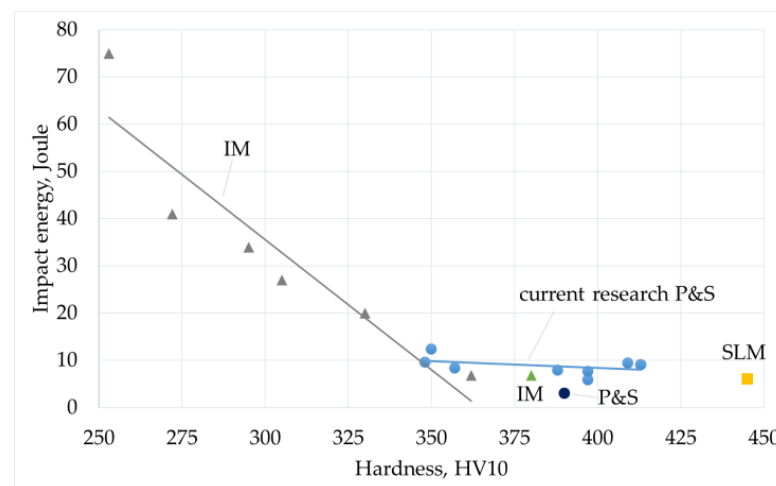


Figure 9. Impact energies of notched 17-4PH SS (H900) specimens in the function of hardness for different manufacturing processes (IM, SLM, and P&S).

Figure 10 shows the morphology of the fractured surfaces taken on samples sintered at different temperatures. As with all the other presented test results, the sintering temperature had a significant effect here as well. In the case of applying the two lower sintering temperatures (1200 °C and 1250 °C), a classic dimple fracture structure can be observed (Figure 10a,b). The only difference between the two cases is that the porosity content is higher in Figure 10a than in Figure 10b.

Further increasing the sintering temperatures (1275 °C) decreases the porosity; however, the fracture mode changes from a dimple to a cleavage fracture (Figure 10c). This is attributed to the increased hardness resulting from the lower porosity. As was highlighted earlier in Figure 4d, inclusions can also be seen at the bottom of almost every dimple on the fracture surfaces.

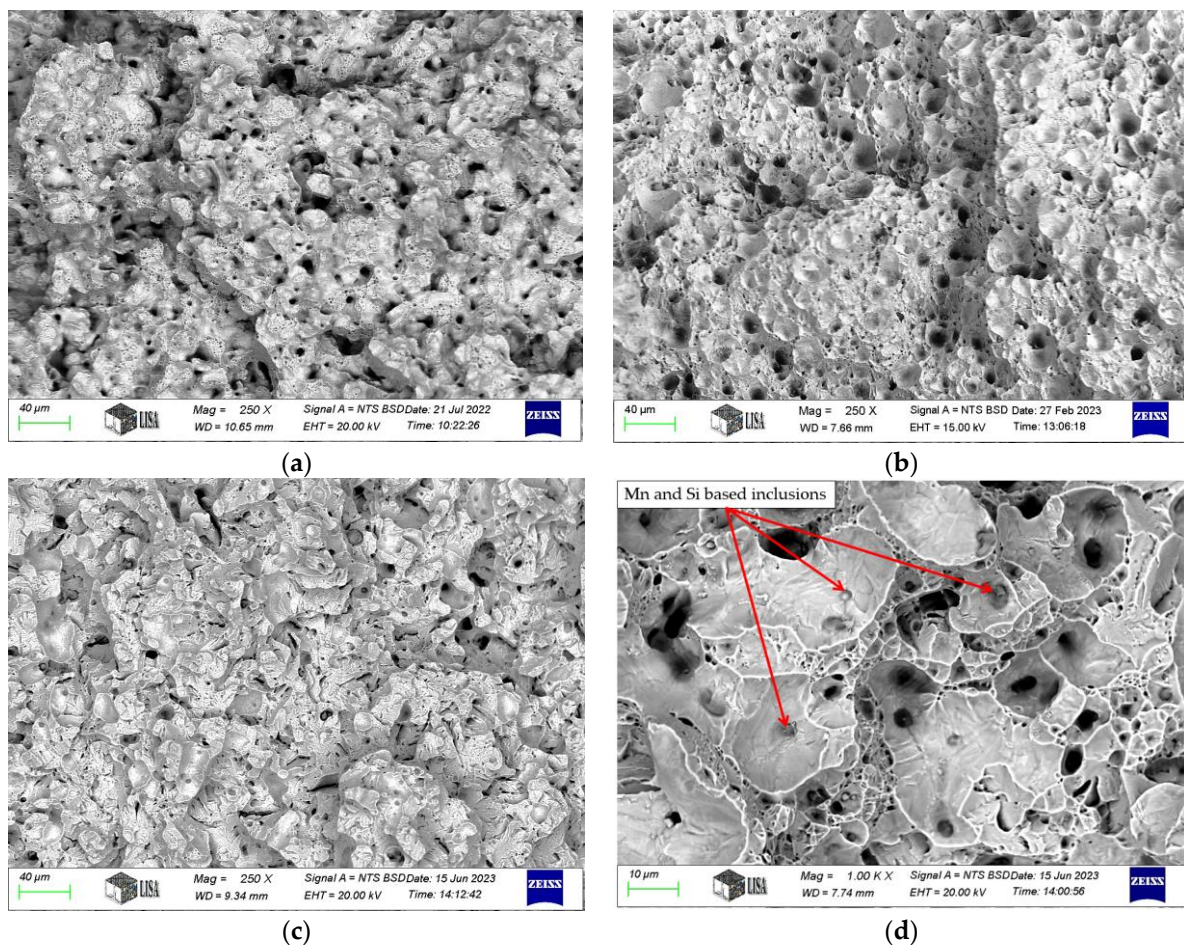


Figure 10. SEM micrographs of the fractured surface of the 17-4PH SS (H900) samples: (a) sintered at 1200 °C for 60 min (250×); (b) sintered at 1250 °C for 60 min (250×); (c) sintered at 1275 °C for 60 min (250×); (d) sintered at 1200 °C for 60 min (1000×).

4. Conclusions

The main goal of the present work was to investigate the effect of the sintering temperature on the properties of 17-4PH stainless steel produced by the classical press and sinter (P&S) method. This type of alloy is not commonly used within the field of conventional powder metallurgy (PM), especially not in its gas-atomized form with spherical morphology. So far, in working with the P&S method, all of the available literature sources reported using water-atomized powder with irregular morphology, and the real novelty of this work was to modify the original spherical morphology to a sub-rounded shape via ball-milling. During ball-milling, the grains changed shape, and their original thin oxide-covered surface also became cleaner and metallurgically more reactive. Due to the so changed geometrical and surface properties, stronger bonds were formed between the adjacent grains during cold pressing. Moreover, applying significantly higher (1.6 GPa) pressure, compared to the conventional levels, the porosity of the green compacts reached down to around 8%. In order to minimize carbon contamination during cold pressing, only the die wall had to be lubricated. As a result of this, degassing was not required, which simplified and accelerated induction sintering. Sintering at 1275 °C for one hour enabled porosity reduction to below 4%, resulting in 1200 MPa yield strength and 1350 MPa ultimate tensile strength with significant (16%) fracture strain. These values are comparable to those of the same alloy products fabricated via ingot metallurgy (IM) or additive manufacturing (AM). Furthermore, the results have shown that there is a clear linear relationship between the porosity content and the investigated properties of the tested material within the applied sintering temperatures (1200 °C and 1275 °C). The research also underscores the importance of optimizing the

manufacturing steps in order to harness the full potential of the 17-4PH SS alloy, ensuring minimal porosity and maximal mechanical integrity for industrial applications.

The results obtained until now are highly encouraging, and some additional fatigue and corrosion tests are also planned to further optimize the P&S manufacturing process.

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