Proceedings of the Pakistan Academy of Sciences: A
Physical and Computational Sciences 60(1): 15-21 (2023)

ISSN (Print): 2518-4245; ISSN (Online): 2518-4253
https://doi.org/10.53560/PPASA(60-1)800

Pakistan Academy of Sciences

Research Article

Temperature-Properties Relationships of Martensitic Stainless Steel for Improved Utilization in Surgical Tools

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Abstract: Sintering temperature and environment plays a very important role in strengthening powder particles of compacting surgical parts by cold powder metallurgy technique. Powder metallurgy is a process of producing components/tools by compacting finely metallic or nonmetallic powders. Generally, in the last decade, these tools were produced by conventional casting techniques but now first time in Pakistan this technique is introduced to develop surgical tools/parts. In this study, the effect of sintering behavior by varying temperatures and environments was studied. The AISI 420 Stainless steel compacted surgical parts (Scalpel and scissor) were sintered at 1000 °C to 1300 °C for 30 minutes in a vacuum and an inert environment in the presence of Argon. The compact density, microstructure and mechanical properties were studied. Microstructural characteristics like porosity, and crystalline size were studied by optical microscope. The hardness values and density of the final parts were also measured through the Rockwell hardness machine and by the Archimedes principle. Decreasing the porosity in the final parts will increase the mechanical properties of sintered parts. Adopting the present process for the development of surgical tools after further refining, the process will prove beneficial in the cost-effectiveness, time and energy saving of the present product.

Keywords: Sintering Temperature, Sintering Environment, Surgical Parts, Stainless Steel, Powder Metallurgy.

1. INTRODUCTION

Powder metallurgy (PM) offers a great advantage when the required criteria for manufacturing are small components in large series with intricate and net shapes [1-3]. PM of martensitic stainless steel is reported to vary on a limited scale due to its difficult processability as compared to other PM of stainless steel [4]. Martensitic starless steel (MSS) and Austenitic stainless steel (ASS) are ordinarily utilized for manufacturing parts with remarkable mechanical and optimum corrosion resistance properties to be worked in different conditions [5, 6]. Their properties can be modified by the heat treatment process, unlike other stainless-steel products [7]. As such, these are widely utilized in dental and surgical instruments, pressure vessels, steam generators, moulds and dies, cutting tools manufacturing as well as automotive sector [8-11].

The PM stainless steel shows limitation in its applications due to low mechanical and corrosion properties as compared to other non-porous materials equivalent to them. Therefore, there is always a primary concern to improve such properties[12, 13]. Enhanced densification by super-solidus liquid phase was attempted by sintering in hydrogen atmosphere for austenitic stainless steel or to increase the mechanical properties such as tensile and fatigue strength by sintering in nitrogen environment and eventually, densification rate was boosted by micro addition of other elements such as silicon. In addition, elements which spark sintering and increase densification such as copper and boron were added to the duplex stainless steel in order to minimize residual porosity and enhancing ductility, so as to improve mechanical behavior of the final samples [12].

Received: January 2023; Accepted: March 2023
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It is proven that sintering-cooling rates have high impact on microstructure of materials [4, 13]. Decrease in corrosion resistance by the presence of brittle phases in austenitic and ferrite stainless steel samples sintered in vacuum and followed by slow cooling. Powder Injection molded austenitic stainless steel (316 L) exhibits improved mechanical and corrosion properties sintered in vacuum with a cooling rate of 10 °C per min, as compared to those cooled at 5 °C per min [14].

Pakistan is one of the major exporters of hand-held surgical instruments throughout the world [15-17]. Owing to the quality of Pakistan's surgical instruments as compared to its cost, various international brands are reported to have shut down their manufacturing units. The surgical industry plays a crucial role as it contributes 1.6 % to overall exports of Pakistan. It has among highly potential items to enhance its contribution to country's overall exports. If proper policymaking is provided, this industry can easily achieve its full potential which will allow it to compete with its global competitors, according to the major industry's players [18, 19].

On the other side of coin, the manufacturers of surgical products in Pakistan are facing several problems. The major problems include lack of adaption of new technology and modernization, continuously decreasing skilled labors and lack of proper R & D for innovation. The industry will face a new and emerging challenge as the European Union is going to introduce Medical Device Regulation (MDR) in the beginning of 2024. According to this law, the surgical instruments will have to be compliant with the new European regulations on biocompatibility. The growing markets of China and Mexico are also posing a major competition for Pakistan's exporter as their innovation sides are more advanced [19-21].

Manufacturing of various engineering products by the use of PM is an emerging field in developing countries. A recent advent 3D printing manufacturing technology, which can be considered as modern shape of Powder Metallurgy is set to revolutionize many industries. 3D printing manufacturing technology has the ability to manufacture any engineering design/parts at a fraction of the present cost. It is expected that this manufacturing technology will change the face of today industries. 3D printing plays a major and important role in the field of medical devices manufacturing [22-24].

Due to the advantage of low labor cost of Pakistani industries, it remains competitive in the international market. The 3D printing manufacturing technology is presently utilized for prototyping only in developed countries, while Pakistan is still sticking to the conventional machining process which uses high energy and human resources. If commercial application of 3D printing started its operation for batch production, our industry might face existential risk in the near future. Therefore, serious steps need to be taken to counter the recent challenges [19].

In the present research, microstructure, density, porosity and mechanical properties of AISI 420 martensitic stainless steel sintered in Argon environment were investigated as a function of temperature for making surgical tools by powder metallurgy, a step way forward to strengthen the manufacturing quality of said tools in Pakistan.

2. MATERIALS AND METHODS

A commercially available AISI 420 martensitic stainless-steel SP-112 grade powder with a mesh size of -200 and zinc stearate as a binder were selected as the base material to prepare surgical tool samples in this research. Powder-binder mixture with different weight percentages of zinc stearate ranges from 0.90 weight % to 4 weight % was prepared in tubular mixer for 30 min. After mixing, zinc stearate powder was homogenously distributed within the stainless-steel powder and hydraulic press of 250 ton's capacity at ~2500 psi for ~8 sec's. Zinc stearate was also used as a die lubricant. Green compact specimens were sintered in Argon atmosphere with a flow rate of Argon gas is 1.5 normal liters per min at different sets of temperature ranges from 1000 °C to 1250 °C, and then cooled in a water-cooled chamber as shown in Table 1. The chemical composition of AISI 420 stainless steel powder is given in Table 2. The schematic representation of the PM process adopted for the present study is shown in Figure 1.

2.1 Characterization Technique

The samples for microstructure evaluation were prepared by cutting the specimen from different parts of surgical tools followed by grinding and polishing. Electrochemical etching using Oxalic acid (10 gram) and distilled water (90 %) was used in order to reveal the microstructure. Density of sintered samples was measured by water

Table 1. Experimental conditions used for sintering of AISI420 stainless steel compacted surgical tools

Sample ID	Base Powder	Binder	Sintering Temperature	Soaking Time	Sintering Environment
Sample 1	420 SS	Zinc stearate	1000 °C	30 min	Argon, Vacuum
Sample 2	420 SS	Zinc stearate	1100 °C	30 min	Argon, Vacuum
Sample 3	420 SS	Zinc stearate	1120 °C	30 min	Argon, Vacuum
Sample 4	420 SS	Zinc stearate	1140 °C	30 min	Argon, Vacuum
Sample 5	420 SS	Zinc stearate	1150 °C	30 min	Argon, Vacuum
Sample 6	420 SS	Zinc stearate	1200 °C	30 min	Argon
Sample 7	420 SS	Zinc stearate	1220 °C	30 min	Argon
Sample 8	420 SS	Zinc stearate	1250 °C	30 min	Argon

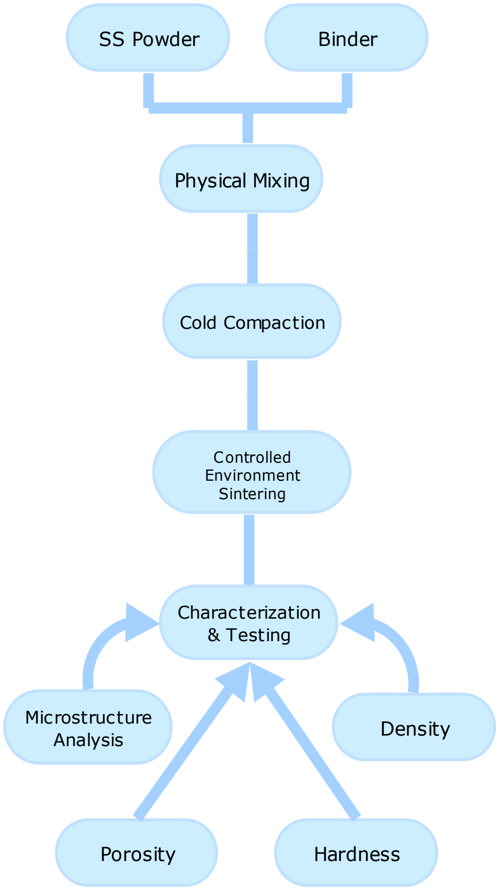

Fig. 1. Schematic representation of Powder Metallurgy Process adopted for present study

Table 2. Composition of AISI 420 martensitic stainless steel as received

Cr	Ni	C	Mo	Mn	Si	Fe
12/14	3/5	0.25-0.35	2/3	≤1	≤1	Balance

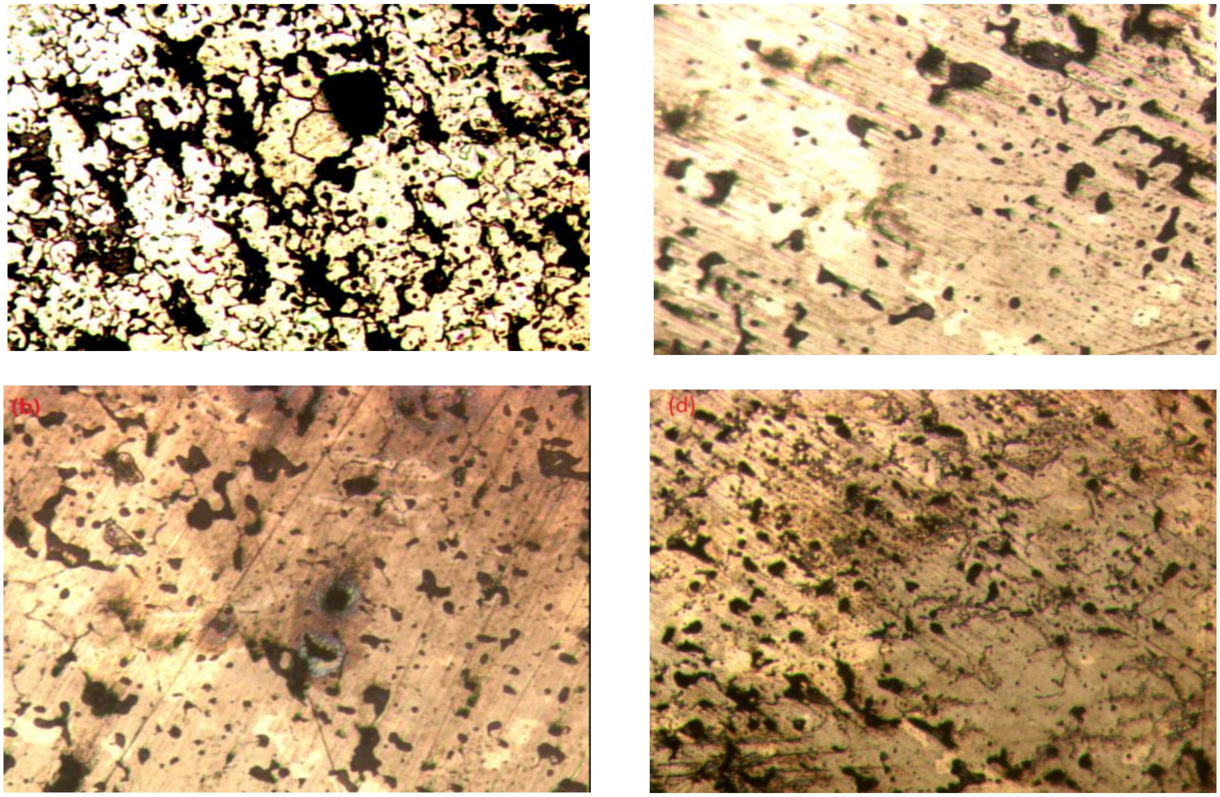

Fig. 2. Optical micrographs of 420 martensitic stainless steel samples sintered at different temperatures (a) 1000 °C, (b) 1150 °C (c) 1200 °C, and (d) 1250 °C

displacement method based on the Archimedes principle in accordance with the ASTM B328-03 standard. Mechanical properties of sintered samples were evaluated using Vickers hardness test. Image J software was utilized to find out the porosity of sintered samples.

3. RESULTS AND DISCUSSION

Microscopic images were obtained in order to examine the porosity of sintered samples shown in Figure 2(a-d). As the samples were sintered within a temperature range of 1000 °C up till 1250 °C under a controlled environment, it was observed that the samples sintered at 1000 °C exhibited higher porosity due to low solid diffusion rate, whereas with the rise in temperature up till 1250 °C, total porosity levels started to decrease. As we know, porosity level can be reduced by two methods in powder metallurgy. The first method is to increase the compaction pressure in order to raise densification of green bodies. The other method is to increase the sintering temperature. At 1100 °C, the porosity level decreases as compared to that of lower temperatures. Upon reaching 1200 °C, the changes in total porosity level becomes prominent, which means proper sintering of green samples has been completed at this temperature, minor or almost negligible changes observed after the temperature at 1250 °C [5, 12] as shown in Figure 3. Moreover, as the sintering temperature increases to around 1200 °C, pore sizes are getting smaller and smaller due to the effect of solid-state diffusion within the samples.

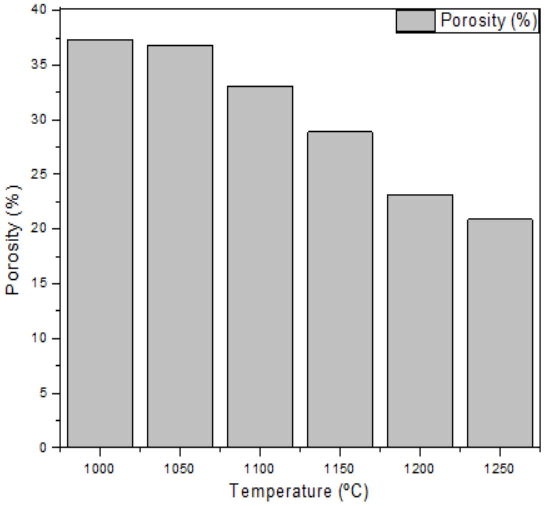

Fig. 3. Percentage/Volume of porosity determined for 420 martensitic stainless steel obtained by Image J

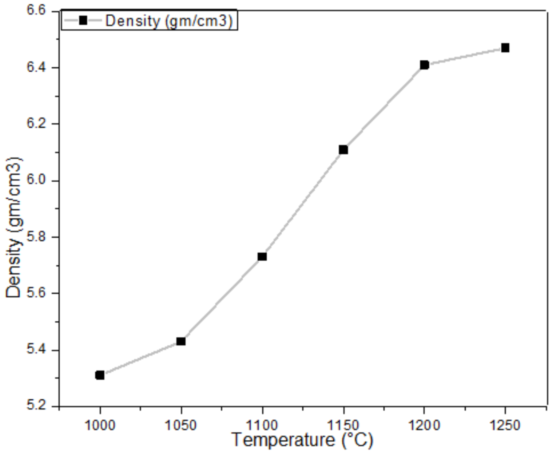

Fig. 4. Density changes with respect to sintering

The sintering process is actually based on diffusion phenomena. The micrographs (Figure 2) clearly show that half of the pores are so small at higher sintering temperature that they act as closed pores as compared to open ones, which are dominant in lower sintering temperature. Relative density of sintered samples was measured by the Archimedes principle. It is clear from Figure 4 that density increases by increasing sintering temperature [25]. As previously discussed, diffusion phenomenon is dominant at higher temperatures, so we obtained dense samples at those temperatures [5]. Hardness of any sintered material strongly depends upon the porosity level percentage. The main idea of our research is to replace surgical tools manufacturing through forging and machining by production through sintering via powder metallurgy. Hardness of sintered samples is always lower compared to those of forged ones. Hardness values (HV) of samples sintered at 1000 °C are about 85 HV, but, at higher sintering temperature, hardness values start to increase up to 205 HV at 1250 °C as shown in Figure 5. Compaction force and cooling rate also affects hardness values and it is very important factor for stainless steel products [26]. Consequently, in this research, cooling of sintered samples in a water-cooled furnace is responsible for increment in hardness values. The final surgical tools developed as a result of present study are presented in Figure 6.

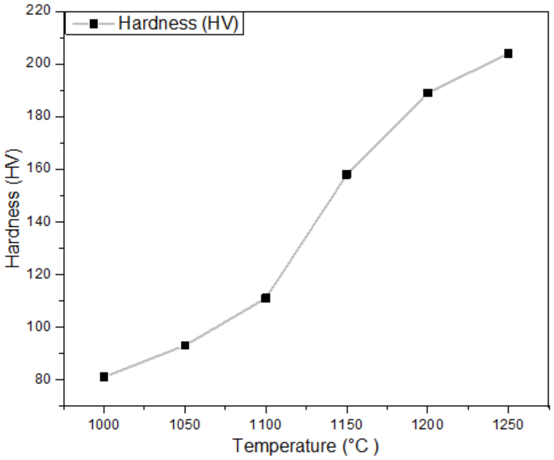

Fig. 5. Comparison of hardness values for different samples

Fig. 6. Surgical tools prepared via powder metallurgy temperature calculated by Archimedes principle

4. CONCLUSION AND RECOMMENDATION

For PM stainless steel, the hardness, pore size and density were found improved by the application of proper sintering temperature and compaction pressure. At high temperature, the diffusion phenomenon becomes high as compared to that at lower temperatures, and it is the main concept behind good mechanical properties and density of the sintered samples. In the present research, unfortunately, we didn't achieve the optimum mechanical properties necessary for commercial surgical tools. However, it was a first step/struggle towards upgradation of aforementioned tools manufacturing process in Pakistan. The present study will open new doors for researchers to work for the betterment in the said field.

Mechanical properties, density and porosity will expectedly increase by sintering of surgical tools in nitrogen and hydrogen environment and we are further working on similar sintering environment. Enhancement of overall properties can expectedly be done by using additive manufacturing process or the novel 3D printing process.

5. CONFLICT OF INTEREST

The authors declare no conflict of interest.

6. REFRENCES

1. G.Y. Liu, B.R. Sun, C.C. Du, S. Li, S.W. Xiu, and T.D. Shen. Hierarchically structured powder metallurgy austenitic stainless steel with exceptional strength and ductility. Materials Science and Engineering: A 861: 144351 (2022).
2. F. Martin, C. Garcia, and M. Aparicio. Tribocorrosion behaviour of powder metallurgy duplex stainless steels sintered in nitrogen. Tribology International 57: 76-85 (2013).
3. M. Javanbakht, M.J. Hadianfard, and E. Salahinejad. Microstructure and mechanical properties of a new group of nanocrystalline medical-grade stainless steels prepared by powder metallurgy. Journal of Alloys and Compounds 624: 17-21 (2015).
4. K. Saeidi, D.L. Zapata, F. Lofaj, L. Kvetkova, J. Olsen, Z. shen, and F. Akhtar. Ultra-high strength martensitic 420 stainless steel with high ductility. Additive Manufacturing 29: 100803 (2019).
5. M. Javanbakht, E. Salahinejad, and M.J. Hadianfard. The effect of sintering temperature on the structure and mechanical properties of medical-grade powder metallurgy stainless steels. Powder Technology 289: 37-43 (2016).
6. S.O. Akinwamide, O.J. Akinribide, M. Mafafo, P. Mjwana, B.A. Obadele, B.T. Abe, and P. Olubambi. Effect of TiN addition on corrosion behavior of spark plasma sintered 304L austenitic stainless steel in chloride media. Materials Today: Proceedings 28: 384-387 (2020).
7. A.N. Acar, A.K. Ekşi, and A. Ekicibil. Effect of pressure on the magnetic and structural properties of X2CrNiMo17-12-2 austenitic stainless steel prepared by powder metallurgy method. Journal of Molecular Structure 1198: 126876 (2019).
8. K.V.V. Nagaraju, S. Kumaran, and T.S. Rao. Electrochemical behavior of various grade P/M stainless steels processed by rapid microwave hybrid sintering (super-solidus) method. Materials Letters 302: 130394 (2021).
9. L.H. Heidari, M.J. Hadianfard, A.R. Khalifeh, D. Vashaee, and L.Tayebi. Fabrication of nanocrystalline austenitic stainless steel with superior strength and ductility via binder assisted extrusion method. Powder Technology 379: 38-48 (2021).
10. S. Pandya, S.R. Kandala, A.R. Annamalai, and A. Upadhaya. Effect of sintering temperature on the mechanical and electrochemical properties of austenitic stainless steel. Materials Science and Engineering: A 556: 271-277 (2012).
11. W. Shen, B. Nan, W. Wang, L. Yu, Q. Zhang, Y. He, X. Huang, and G. yuan. In situ synthesis and strengthening of ultra high-carbon martensitic stainless steels in addition of LaB6. Journal of Alloys and Compounds 738: 363-371 (2018).
12. F. Martín, C. Garcia, Y. Blanco, and M. L. R-Mendez. Influence of sinter-cooling rate on the mechanical properties of powder metallurgy austenitic, ferritic, and duplex stainless steels sintered in vacuum. Materials Science and Engineering: A 642: 360-365 (2015).
13. C. García, F. Martin, and Y. Blanco. Effect of sintering cooling rate on corrosion resistance of powder metallurgy austenitic, ferritic and duplex stainless steels sintered in nitrogen. Corrosion Science 61: 45-52 (2012).
14. M.R. Raza, F. Ahmad, M.A. Omar, and R.M. German. Effects of cooling rate on mechanical properties and corrosion resistance of vacuum sintered powder injection molded 316L stainless steel. Journal of Materials Processing Technology 212(1): 164-170 (2012).
15. M.F. Bhutta. Quality of Surgical Instruments. The Annals of The Royal College of Surgeons of England 89(2): 190-190 (2007).
16. A. Ikram, Q. Su, and M. Sadiq. Technical Efficiency And Its Determinants: An Empirical Study Of Surgical Instruments Cluster Of Pakistan. Journal of Applied Business Research 32: 647-660 (2016).
17. M. Junaid, R.N. Malik, and D.-S. Pei. Health hazards of child labor in the leather products and surgical instrument manufacturing industries of Sialkot, Pakistan. Environmental Pollution 226: 198-211 (2017).
18. M.Z. Khan. Pakistan's shadow surgical instruments' sector. Report, Dawn/22.08, Business & Finance weekly (2016). https://www.dawn.com/news/1279191(accessed 1 January 2023).
19. S.S. Amir, and S. Arshad. Enhancing the competitiveness of pakistan's surgical instruments industry. Report PBC/EDB/SIMAP 1-79 (2022).
20. U.A. Mufti. The Surgical Instruments Industry

of Pakistan is World #1 in Production. Report Bussiness/28.04, Economy.pk (2022). https://www.economy.pk/the-surgical-instruments-industry-of-pakistan/ (accessed 1 January 2023).

21. C. Berman. Labor standards in Pakistan's surgical instruments sector: a synthesis report. Ethical Trading Initiative (ETI) UK.
22. A. Sola, A. Trinchi, and A.J. Hill. Self-assembly meets additive manufacturing: Bridging the gap between nanoscale arrangement of matter and macroscale fabrication. Smart Materials in Manufacturing 1: 100013 (2023).
23. M. Armstrong, H. Mehrabi, and N. Naveed. An overview of modern metal additive manufacturing technology. Journal of Manufacturing Processes 84: 1001-1029 (2022).
24. M. Nishal, K.R. Prasad, M.S. Dashageer and A.G. Ragunath. Significance of additive manufacturing amidst the pandemic. Materials Today: Proceedings 72: 2540–2546 (2022).
25. D. Cui, and J. Wang. Research on Powder Metallurgy High Nitrogen Stainless Steels. Advanced Materials Research 690-693: 172-175 (2013).
26. F. Martín, C. Garcia, Y. Blanco, and M.L. R-Mendez. Influence of sinter-cooling rate on the mechanical properties of powder metallurgy austenitic, ferritic, and duplex stainless steels sintered in vacuum. Materials Science and Engineering: A 642: 360-365 (2015).