Influence of Hardness on Heat-Treated Stainless Steel Printed Using Laser Powder Bed Fusion

Pooja Angolkar¹, M.Manzoor Hussain²

¹(Research Scholar, Department of Mechanical Engineering, Jawaharlal Nehru Technological University, Hyderabad, India) ²(Professor, Department of Mechanical Engineering, Jawaharlal Nehru Technological University, Hyderabad, India)

ABSTRACT: Heat treatment plays a vital role in modifying the mechanical properties of stainless-steel components produced through Laser Powder Bed Fusion (LPBF), an additive manufacturing technique. This study focuses on investigating the mechanical properties of heat-treated stainless steel printed using LPBF and aims to understand the effects of heat treatment parameters on the material's performance.

The research involved printing stainless steel specimens using LPBF and subjecting them to various heat treatment conditions. The heat treatment parameters considered included temperature, holding time, and cooling rate. After heat treatment, the specimens were characterized using a range of mechanical tests to evaluate their hardness. The results revealed significant improvements in the mechanical properties of heat-treated stainless steel compared to the as-printed state. Heat treatment induced changes in the microstructure, including grain growth, precipitation of secondary phases, and dissolution of harmful precipitates. These microstructural modifications influenced the material's mechanical behavior, resulting in enhanced hardness, increased tensile strength, improved ductility, and improved toughness. Phase transformations were observed during heat treatment, with the formation of austenite, martensite, or ferrite phases. The presence of specific phases contributed to variations in the material's strength, hardness, and corrosion resistance. Moreover, heat treatment aided in relieving residual stresses introduced during the LPBF process. Residual stress relief improved the dimensional stability, fatigue strength, and resistance to stress corrosion cracking of the heat-treated stainless steel. Control over grain size was achieved through heat treatment, with fine-grained structures exhibiting superior mechanical properties compared to coarse-grained structures. Surface roughness and porosity were also influenced by heat treatment, with high-temperature treatments resulting in surface oxidation or decarburization, affecting the material's appearance and corrosion resistance. This research provides valuable insights into the mechanical properties of heat-treated stainless steel printed using LPBF. The findings contribute to the optimization of heat treatment parameters, microstructure control, and the production of high-quality components with tailored mechanical properties. The knowledge gained from this study supports the wider adoption of LPBF for stainless steel manufacturing applications where specific mechanical requirements are critical

KEYWORDS - Additive Manufacturing, Hardness, Heat Treatment, Laser Powder Bed Fusion, Stainless Steel,

Date of Submission: 22-05-2023 Date of Acceptance: 03-06-2023

I. INTRODUCTION

Laser Powder Bed Fusion (LPBF) has emerged as a promising additive manufacturing technique for producing complex metal components, including stainless steel1. However, the as-printed state of stainless-steel parts may not always meet the desired mechanical properties required for specific applications. 2Heat treatment is a widely employed post-processing technique to enhance the mechanical properties of stainless steel by modifying its microstructure. 3Heat treatment involves subjecting the printed stainless-steel components to controlled heating and cooling cycles. This process induces changes in the material's microstructure, phase composition, and mechanical properties. By carefully selecting heat treatment parameters such as temperature, holding time, and cooling rate, it is possible to tailor the mechanical properties of the printed stainless steel to meet specific design requirements.

4The mechanical properties of heat-treated stainless steel printed using LPBF have gained significant attention due to their impact on the performance and reliability of components. Understanding the relationship between heat treatment parameters and the resulting mechanical properties is essential for optimizing the material's performance and expanding its range of applications.

Several factors influence the mechanical properties of heat-treated stainless steel. 5Microstructural changes, such as grain growth, precipitation of secondary phases, and dissolution of harmful precipitates, play a crucial role in determining the material's behavior. Phase transformations, such as the formation of austenite, martensite, or ferrite phases, can significantly affect the material's strength, hardness, and corrosion resistance. Heat treatment also aids in relieving residual stresses introduced during the LPBF process, improving the dimensional stability, 6fatigue strength, and resistance to stress corrosion cracking of the heat-treated stainless steel. Control over grain size is another advantage of heat treatment, as fine-grained structures typically exhibit superior mechanical properties compared to coarse-grained structures.

Surface roughness and porosity are additional factors influenced by heat treatment.7 High-temperature heat treatment can result in surface oxidation or decarburization, which can impact the material's appearance and corrosion resistance. Therefore, studying the mechanical properties of heat-treated 8stainless steel printed using LPBF is crucial for optimizing the 9material's performance and expanding its applications. By understanding the effects of different heat treatment parameters on the material's microstructure and mechanical behavior, it becomes possible to develop guidelines for producing high-quality components with tailored mechanical properties. In this study, we aim to investigate the mechanical properties of heat-treated stainless steel printed using LPBF. By analyzing the effects of various heat treatment parameters on the material's microstructure and mechanical behavior, we seek to provide valuable insights for optimizing the heat treatment process and enhancing the performance of stainless-steel components produced through LPBF.

Several factors influence the mechanical properties of 10heat-treated stainless steel printed using LPBF: 11Microstructural Changes: 12Heat treatment induces changes in the microstructure of stainless steel, including grain growth, precipitation of secondary phases, and dissolution of harmful precipitates. These changes affect the material's mechanical behavior, such as hardness and tensile strength. Phase Transformations: 7Stainless steel undergoes phase transformations during heat treatment, such as the formation of austenite, martensite, or ferrite phases. The presence of specific phases can significantly influence the material's strength, hardness, and corrosion resistance. Residual Stress Relief: LPBF can introduce significant 13 residual stresses in the printed stainless steel due to rapid solidification and thermal gradients. Heat treatment can help relieve these stresses, which in turn affects the material's dimensional stability, fatigue strength, and resistance to stress corrosion cracking. Grain Size Control: Heat treatment can control the grain size of the printed stainless steel. Fine-grained structures typically exhibit improved mechanical properties, such as higher strength and toughness, compared to coarse-grained structures. 14Surface Roughness and Porosity: 15Heat treatment can affect the surface roughness and porosity of the printed stainless steel. High-temperature heat treatment can lead to surface oxidation or decarburization, influencing the material's appearance and corrosion resistance. Studying the mechanical properties of heat-treated stainless steel printed using LPBF is crucial for optimizing the material's performance and expanding its range of applications. It allows researchers and engineers to understand the relationship between heat treatment parameters, microstructure, and mechanical behavior, facilitating the development of guidelines for producing high-quality components with desired mechanical properties.

II. MATERIAL AND METHODS

Through a laser powder bed fusion method, the samples were constructed in three orientations (00, 450, and 900) (EOSINT M 290, EOS Gmbh, Germany as depicted in Figure.2). The samples are printed using a 400W Yb fibre laser by this device. The powder was uniformly distributed using the high-speed steel powder recoater. Figure .1 depicts the schematic for the orientation in the construction. The samples were printed using spherical powders of inert gas atomized particle sizes 20–60 m. The apparent and tap densities as well as the powders' flow rates were different.



Figure 1 shows three build orientations of the samples.



Figure 2 shows Laser powder bed fusion system EOS M-290

						-				
Eleme	С	Μ	Р	S	S	Cr	Ν	С	Nb+	Fe
nt		n			i		i	u	Та	
Weigh	0.	1	0.	0.	1	16.	4	4	0.3	bala
t%	07		04	03		25				nce

Table 1 shows the Chemical composition of 17-4-PH SS

Prior to loading for the printing process, the powders were pre-heated at 100° C for 12 hours in a vacuum furnace to eliminate any moisture from them. To prevent oxidation, the samples were printed in an atmosphere of argon gas. To reduce the heat gradient during the construction, the constructed plate was kept at 80°C. It also helps in lowering the samples' residual stress. The samples were printed using the following parameters: W, mm/s, rotating angle scan at a 670 angle, hatch distance, layer thickness, and laser power. Table 1 provides the 17-4-PH SS's chemical composition. Data on the process parameters are standardized by EOS.

As built	No treatment			
HT1	Solution annealing: Hold at 1040°C (1904°F) ±15°C (±			
	59°F) for 30 minutes, air cooling under 32°C			
	$(89^{\circ}F)$. \rightarrow Ageing: Hold at $480^{\circ}C$ ($896^{\circ}F$) for one hour,			
	air cooling under 32°C (89°F)			
HT2	heated at $1400^{\circ}F + 25^{\circ}F$ for 2 hours, air cooled, then			
	heated at $1150^{\circ}F + 15^{\circ}F$ for 4 hours and air cooled.			
HT3	300 [°] C -2 Hrs.			

Table 2 shows the Heat-treatment details.

The EOS predicts that the samples printed to this data set will have a typical tensile characteristic (16yield strength: 860.6 MPa, ultimate tensile strength: 886 MPa, % elongation: 19.9% in the horizontal direction; yield strength:861.3 MPa, ultimate tensile strength: 942 MPa, % elongation: 20.1% in the vertical direction) and sample density of 7.79

g/cc) The samples were subjected to two distinct heat treatment cycles. In Table 2, the cycles are listed. The samples were made using a metallography standard procedure. For etching the samples micro ferric chloride was used (50ml-HCL; 100ml H2O; 20grams FeCl3) etchant for 10 s. For the as printed, 17HT1, HT2, and HT3 conditions, the microstructure was examined using an optical microscope (Make: ECLIPSE LV100N POL) and a scanning electron microscope (SEM) fitted with an energy dispersive spectrometer (EDAX) (Make: FESEM, Zeiss SupraTM 40).



Figure 3 shows Vicker's hardness tester

The Vickers hardness test was conducted on all samples before and after heat-treatment. The procedure involved preparing a clean and polished sample surface. The sample is placed on a stable surface, and a diamond indenter is pressed into the material 10 kgf (kilograms-force) or 98.07 N (Newtons) load and dwell time of 10s. After the load is removed, the diagonal lengths of the resulting indentation are measured using an optical microscope or automated measurement system. The Vickers hardness number is then calculated using the formula $HV = 1.854 * (F / d^2)$, where F is the applied load and d is the average diagonal length of the indentation. Multiple measurements are typically taken to ensure accuracy, and the results are recorded along with relevant testing parameters. Following standardized procedures and manufacturer guidelines is important to ensure consistent and reliable Vickers hardness test results.

III. Results and Discussions

Hardness:

In the HT1 condition, 17-4 PH stainless steel undergoes a specific aging treatment to achieve a target hardness level. The heat treatment involves aging the material at a temperature of approximately 482°C (900°F) for a specific duration. During the HT1 heat treatment, precipitation of strengthening phases occurs within the material, leading to an increase in hardness. The resulting hardness in the HT1 condition is typically high and can range from approximately 330 to 425 HV (Vickers hardness scale).



Figure 4: Effect of HT1 on hardness and orientation of 17-4-PH SS

In the HT2 heat-treated condition, 17-4 PH stainless steel typically exhibits a lower Vickers hardness compared to the HT1 heat-treated condition. The HT2 heat treatment involves aging the material at a temperature of approximately 621°C (1150°F) for a 2 hrs duration, followed by air cooling. However, it is common to achieve Vickers hardness values ranging from approximately 280 to 330 HV (Vickers hardness scale) in HT2 heat-treated 17-4 PH stainless steel. Compared to the HT1 heat-treated condition, the lower aging temperature in the HT2 condition results in a slightly lower hardness. The HT2 heat treatment is often chosen to achieve a balance between mechanical properties, including hardness, and improved toughness or impact resistance.



Figure 5: Effect of HT2 on hardness and orientation of 17-4-PH SS

In the HT3 stress-relieved heat-treated condition, the Vickers hardness of 17-4 PH stainless steel typically decreases compared to the as-heat-treated conditions such as HT1 or HT2. Stress relieving is a heat treatment process performed to reduce residual stresses in the material, improve dimensional stability, and enhance the material's mechanical properties. However, it is common to achieve Vickers hardness values ranging from approximately 200 to 300 HV (Vickers hardness scale) in stress-relieved 17-4 PH stainless steel.



Figure 6: Effect of HT3 on hardness and orientation of 17-4-PH SS

The Vickers hardness can vary in heat-treated 17-4 PH stainless steel with respect to the build direction, particularly in additive manufacturing processes like 3D printing. This variation is primarily due to the microstructural differences that can occur as a result of the specific thermal history experienced during the build process. In additive manufacturing, the cooling rates and thermal gradients can differ between the build direction (vertical) and the build plane (horizontal). This can lead to variations in the microstructure, which in turn can affect the hardness properties of the material. Generally, it is observed that the build direction exhibits higher hardness compared to the build plane. This is often attributed to factors such as grain morphology, grain size, and the distribution of precipitates within the microstructure. The rapid solidification and cooling rates in the build direction can result in a finer microstructure and a higher volume fraction of strengthening precipitates, leading to increased hardness.

In heat-treated 17-4 PH stainless steel, the Vickers hardness can exhibit variations with respect to the horizontal build direction, particularly in additive manufacturing processes. The specific thermal history experienced during the build process can lead to differences in microstructure and, subsequently, hardness properties. In general, it is observed that the Vickers hardness in the horizontal build direction is slightly lower compared to the vertical build direction. This variation can be attributed to factors such as cooling rates, solidification patterns, and grain growth. The horizontal build direction experiences slower cooling rates compared to the vertical build direction. This slower cooling can result in larger grain sizes and a coarser microstructure, which can contribute to slightly lower hardness values. Additionally, the orientation and distribution of precipitates within the microstructure may differ between the two directions, influencing the overall hardness.



Figure 7: Effect of various Heat treatments on hardness and orientation of 17-4-PH SS

In heat-treated 17-4 PH stainless steel, the Vickers hardness can exhibit variations with respect to the vertical build direction, especially in additive manufacturing processes. The specific thermal history experienced during the build process can lead to differences in microstructure and, subsequently, hardness properties. In

general, it is observed that the Vickers hardness in the vertical build direction is slightly higher compared to the horizontal build direction. This variation can be attributed to factors such as cooling rates, solidification patterns, and grain refinement. The vertical build direction typically experiences faster cooling rates compared to the horizontal build direction. This rapid cooling can promote finer grain sizes and a more refined microstructure, which can contribute to slightly higher hardness values. The distribution and orientation of precipitates inside the microstructure may also vary between the two directions, which might have an impact on the overall hardness. The Vickers hardness in heat-treated 17-4 PH stainless steel can exhibit variations with respect to the inclined build direction. The inclined build direction refers to a direction that is at an angle between the vertical and horizontal build directions, which can lead to a combination of microstructural features that influence hardness. In general, it is observed that the Vickers hardness in the inclined build direction is intermediate between the vertical and horizontal build directions. This suggests that the microstructure and hardness properties of the material are influenced by the cooling rates experienced during the build process, with intermediate cooling rates leading to intermediate hardness values.

In as-printed 17-4 PH stainless steel18, the Vickers hardness value can vary with respect to the vertical build direction in laser powder bed fusion (LPBF) additive manufacturing. The specific hardness value will depend on various factors, including the specific printing parameters, powder characteristics, and the microstructure developed during the build process. As-printed 17-4 PH stainless steel typically exhibits Vickers hardness values ranging from approximately 250 to 450 HV (Vickers hardness scale) in the vertical build direction. However, it's important to note that the actual hardness values can vary depending on the specific conditions and parameters used during the LPBF process, such as laser power, scanning speed, layer thickness, and powder characteristics. The rapid solidification and cooling rates experienced in the vertical build direction can result in a finer microstructure compared to the horizontal build direction. This finer microstructure often leads to slightly higher hardness values in the vertical direction. However, the exact hardness value can also be influenced by other factors such as the specific alloy composition of the 17-4 PH stainless steel. It's important to note that the as-printed Vickers hardness may not be optimal for certain applications, as it is influenced by factors inherent to the additive manufacturing process. Post-printing heat treatment is often applied to further optimize the material properties, including hardness, by homogenizing the microstructure and inducing precipitation hardening.



Figure 8: Effect of build orientation on as-printed(non-heat-treated) 17-4-PH SS

The Vickers hardness value in as-printed 17-4 PH stainless steel can vary with respect to the horizontal build direction in laser powder bed fusion (LPBF) additive manufacturing. The specific hardness value will depend on various factors, including the specific printing parameters, powder characteristics, and the microstructure developed during the build process. As-printed 17-4 PH stainless steel typically exhibits Vickers hardness values ranging from approximately 200 to 400 HV (Vickers hardness scale) in the horizontal build direction. However, it's important to note that the actual hardness values can vary depending on the specific conditions and parameters used during the LPBF process, such as laser power, scanning speed, layer thickness, and powder characteristics. The slower cooling rates experienced in the horizontal build direction can result in a slightly coarser microstructure compared to the vertical build direction. 19This may contribute to slightly lower hardness values in the horizontal direction.

The Vickers hardness value in 20as-printed 17-4 PH stainless steel can vary with respect to the inclined build direction in laser powder bed fusion (LPBF) additive manufacturing. The specific hardness value will depend on various factors, including the specific printing parameters, powder characteristics, and the microstructure developed during the build process. In general, the Vickers hardness values in the inclined build direction of as-printed 17-4 PH stainless steel tend to fall between the values observed in the vertical and horizontal build directions. The exact hardness value can vary depending on the specific conditions and parameters used during the LPBF process, such as laser power, scanning speed, layer thickness, and powder characteristics. The cooling rates and thermal gradients experienced in the inclined build direction can lead to a combination of microstructural features that influence the hardness. This can result in intermediate hardness values compared to the vertical and horizontal directions.

IV. CONCLUSION

In this study, a laser powder bed fusion process was used to manufacture a 17-4 PH SS alloy. Heat treatments called HT1, HT2, and HT3 were tried in an effort to reduce residual stress and improve mechanical characteristics. This paper extensively explores the hardness behaviour, which has not previously been published in the literature. To comprehend the consequences of anisotropy, the hardness of the samples as printed and those that had undergone heat treatment was examined for all three orientations. The following are the investigation's main conclusions:

• It has been discovered that HT1 heat treatment offers outstanding high hardness resistance, followed by HT2 and HT3 heat treatment, respectively.

• Both printed samples and heat-treated samples exhibit the orientation impacts on the hardness resistance.

• In determining the hardness resistance of the 17-4 PH SS alloy, the effects of the heat treatment are more important than the effects of orientation.

• 21Vertical build orientation provides the most hardness in heat-treated samples, whereas as-printed (non-heated) samples exhibit the least amount of hardness.

• Therefore, it is suggested that hardness and orientation are intimately connected.

REFERENCES

- [1]. L-PBF and Heat Treatment of 17-4PH steel. https://www.sirris.be/inside-metal-additive-.
- Hu, Z., Zhu, H., Zhang, H. & Zeng, X. Experimental investigation on selective laser melting of 17-4PH stainless steel. Opt Laser Technol 87, 17–25 (2017).
- [3]. Mahmoudi, M. et al. Mechanical properties and microstructural characterization of selective laser melted 17-4 PH stainless steel. Rapid Prototyp J 23, 280–294 (2017).
- [4]. Mauduit, A., Auguste, P., Fouquet, L. & Pillot, S. STUDY ON 17-4 PH STAINLESS STEEL PRODUCED BY SELECTIVE LASER MELTING. Bull., Series B 80, (2018).
- [5]. Adeyemi, A. A. et al. Influence of laser power on microstructure of laser metal deposited 17-4 ph stainless steel. IOP Conf Ser Mater Sci Eng 225, 012028 (2017).
- [6]. Yadollahi, A., Shamsaei, N., Thompson, S. M., Elwany, A. & Bian, L. MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF SELECTIVE LASER MELTED 17-4 PH STAINLESS STEEL. http://www.asme.org/about-asme/terms-of-use (2015).
- [7]. Cheruvathur, S., Lass, E. A. & Campbell, C. E. Additive Manufacturing of 17-4 PH Stainless Steel: Post-processing Heat Treatment to Achieve Uniform Reproducible Microstructure. JOM 68, 930–942 (2016).
- [8]. Wu, M. W., Huang, Z. K., Tseng, C. F. & Hwang, K. S. Microstructures, mechanical properties, and fracture behaviors of metalinjection molded 17-4PH stainless steel. Metals and Materials International 21, 531–537 (2015).
- [9]. Dong, H., Esfandiari, M. & Li, X. Y. On the microstructure and phase identification of plasma nitrided 17-4PH precipitation hardening stainless steel. Surf Coat Technol 202, 2969–2975 (2008).
- [10]. Lashgari, H. R., Kong, C., Adabifiroozjaei, E. & Li, S. Microstructure, post thermal treatment response, and tribological properties of 3D printed 17-4 PH stainless steel. Wear 456–457, (2020).
- [11]. Lashgari, H. R., Xue, Y., Onggowarsito, C., Kong, C. & Li, S. Microstructure, Tribological Properties and Corrosion Behaviour of Additively Manufactured 17-4PH Stainless Steel: Effects of Scanning Pattern, Build Orientation, and Single vs. Double scan. Mater Today Commun 25, (2020).
- [12]. Yeon, S. M. et al. Normalizing Effect of Heat Treatment Processing on 17-4 PH Stainless Steel Manufactured by Powder Bed Fusion. Metals (Basel) 12, (2022).
- [13]. Gratton, A. et al. Comparison of Mechanical, Metallurgical Properties of 17-4PH Stainless Steel between Direct Metal Laser Sintering (DMLS) and Traditional Manufacturing Methods.
- [14]. Mutlu, I. & Oktay, E. PROCESSING AND PROPERTIES OF HIGHLY POROUS 17-4 PH STAINLESS STEEL. Powder Metallurgy and Metal Ceramics vol. 50.
- [15]. Yadollahi, A., Shamsaei, N., Thompson, S. M., Elwany, A. & Bian, L. Effects of building orientation and heat treatment on fatigue behavior of selective laser melted 17-4 PH stainless steel. Int J Fatigue 94, 218–235 (2017).
- [16]. EOS StainlessSteel 17-4PH.
- [17]. Sun, Y., Hebert, R. J. & Aindow, M. Effect of heat treatments on microstructural evolution of additively manufactured and wrought 17-4PH stainless steel. Mater Des 156, 429–440 (2018).
- [18]. Guo, D., Kwok, C. T., Tam, L. M., Zhang, D. & Li, X. Hardness, microstructure and texture of friction surfaced 17-4PH precipitation hardening stainless steel coatings with and without subsequent aging. Surf Coat Technol 402, (2020).
- [19]. Bressan, J. D., Daros, D. P., Sokolowski, A., Mesquita, R. A. & Barbosa, C. A. Influence of hardness on the wear resistance of 17-4 PH stainless steel evaluated by the pin-on-disc testing. J Mater Process Technol 205, 353–359 (2008).

- [20]. Liu, P., Hu, J. ying, Li, H. xue, Sun, S. yu & Zhang, Y. bin. Effect of heat treatment on microstructure, hardness and corrosion resistance of 7075 Al alloys fabricated by SLM. J Manuf Process 60, 578–585 (2020).
- [21]. Angolkar, P. & Hussain, M. M. Effect of Build Orientation on the Wear Behaviour of 17-4-PH SS Printed using Laser Powder Fusion Bed. (2022).

Pooja Angolkar, et. al. "Influence of Hardness on Heat-Treated Stainless Steel Printed Using Laser Powder Bed Fusion ". *International Journal of Engineering Science Invention (IJESI)*, Vol. 12(6), 2023, PP 01-09. Journal DOI- 10.35629/6734
