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Experimental Investigations on Wear and Mechanical Properties of Post Heat Treated AM 17-4 PH SS Parts

Gunjan Chauhan[1], Prof. Niraj Bala[2], Prof. S.S. Dhami[3]
[1, 2, 3]Mechanical Engineering Department, NITTTR Chandigarh

Abstract: Selective laser melting (SLM) is an advanced additive manufacturing (AM) technique which constructs 3D objects by melting powder materials in a laser, layer by layer, according to a computer-aided design (CAD) model. SLM is utilized to create products from a variety of materials, with qualities equivalent to benchmarked conventionally manufactured components. Because of these qualities, 17-4PH stainless steel is a suitable material for a diverse range of industrial applications, including structural parts in the aerospace, chemical and petrochemical sectors. The specimens using 17-4PH stainless steel powder were successfully manufactured by optimizing the parameters using SLM process. The as built specimens were heat treated using two different cooling rates. Present research paper evaluates and compares the microstructure, wear behavior and tensile strength of the finished additively manufactured specimens with as built, post heat treated and wrought specimens.
Keywords: Selective laser melting, 17-4PH stainless steel, Heat treatment, Microstructure, Wear behaviour

I. INTRODUCTION

Additive manufacturing (AM) is an unique process for material incrementation that involves the synthesis of material layer over the layer. This method differs from traditional manufacturing method for subtraction of material where only the required material is kept and the rest is removed off the workpiece surface in form of small chips so as to get a desired form. Additive manufacturing has been receiving an increased focus since a decade because of its inherent benefits, for example exceptional flexibility in design as well as shorter lead times [1-2]. Another very common terminology used in creation of 3D objects in a layered manner is referred as Rapid prototyping. New technologies available today enable the production of items that are far more than prototypes. Rapid manufacturing (RM) is used when it is evident that such technologies that builds prototypes as well as it moulds matrices and tools.[3]. In comparison to RM, RP and 3D Printing, Additive manufacturing is often recognized for being most suitable for characterizing this advanced manufacturing technology's processing technique. Only a few of the known AM processes can manufacture metallic components. In this respect, the most popular techniques are electron beam melting (EBND), Additive manufacturing techniques such as laser sintering and direct energy deposition. It is well-known that LAM is an incremental and layered technology., using powder bed fusion for printing of metals. SLM is one such process that directly fabricates 3D components from metallic powders by melting and solidifying the chosen areas layer using digital model [4-7]. Process parameters including scan speed, laser power, thickness, hatch spacing, and build orientation are adjusted to control the process. To achieve the desired properties, these variables must be optimised. After production is complete, the construction chamber is brushed clean of any stray powders, and then the component is removed from the substrate plate through wire cut electrical discharge machining. The entire process occurs in a nitrogen/argon rich environment which is controlled to preserve the completed product against decarburization, oxidation and other processes that degrade mechanical qualities of the finished components [8]. SLM of aluminum alloy was studied by Loh et al. [9], who looked at how Gaussian and constant beam profiles affected the process. By raising the scan speed and decreasing hatch spacing, a constant laser was able to produce a wider melt pool with less melt penetration. Rashid et al. [11] investigated the scan approach impact on the mechanical characteristics and density of 17-4PH SS material after SLM processing. This investigation involves usage of alternative scan techniques, where one includes scanning every layer once (scan 'O') while the other involves multiple scans of the layer (Scan 'X'). According to the research, the double-scanning method results in manufactured components with greater relative density as well as high hardness than those developed using the single-scan method. Irrinkiet al. [12] carried out an analysis to understand the properties of powder including energy density resulting by increased density along with mechanical behavior of a 17-4PH SS alloy manufactured using SLM. Their observations discovered that an increase in energy density enhances the hardness and tensile strength for both of the water and gas atomized powders.

This discovery proved that it is possible to build components with superior characteristics using SLM from affordable water-atomized powder. According to the current reviewed literature, there are very few systematic studies for evaluating mechanical characteristics for 17-4PH stainless steel produced by SLM. Hence this work attempts to investigate the properties of additively built SLM-produced post heat treated and wrought specimens.

The current study stresses on mechanical and wear behavior of post heat treated 17-4 PH SS specimens manufactured using SLM technique. Post-HT cooling rates have been shown to be a vital factor that has a major effect on the mechanical properties and microstructure of metallic components. Therefore, the goal of the present research aims to find out how different post-annealing cooling rates affect the microstructure, mechanical and wear properties of 17-4 PH SS made by selective laser melting. Further the results have been compared with wrought 17-4PH

II. EXPERIMENTAL WORK

A. Sample Fabrication

The raw material for this experiment is 17-4PH (Alloy 630) stainless steel which is vaporized by gas provided by 3D systems. The foundation material for the studies was 17-4PH SS powder ranging in size from 20 to 40 microns. To avoid oxidation and contamination, the powder is produced in an inert gas medium which is usually nitrogen. Table 1 lists the powder's chemical composition.

TABLE I CHEMICAL COMPOSITION OF 17-4 PH SS POWDER

Elements	Cr	Ni	Cu	Si	Ta + Nb	Mn	Fe
Weight (In %)	17.45	4.00	4.10	0.60	0.30	0.40	73.15

EDS with a scanning electron microscope (SEM), model JEOL-IT100, was used to determine the elements present in the composition of the chosen powder and the almost spherical shape of the powder desired for SLM has been depicted in Fig. 1

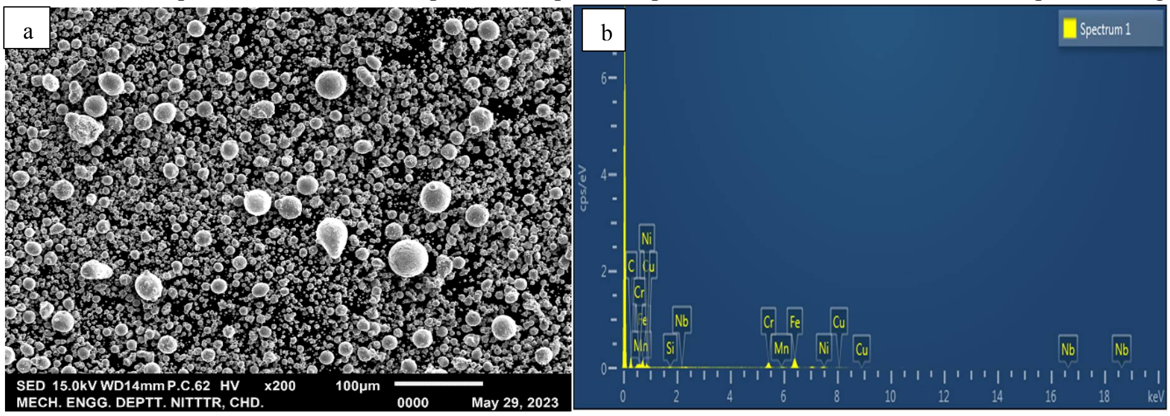

Fig. 1 (a) SEM image of powder and (b) EDS of powder

A SLM machine was used to fabricate 3D forms out of 17-4PH Stainless Steel powder (ProX DMP 200 from 3D Systems) volume dimensions of 140 mm x 140 mm x 115 mm. The samples were constructed using an SS 430F substrate plate. Process parameters were set as per manufacturer's recommendation to achieve better overall characteristics. As shown in Table 2, the operating process parameters combination that generates denser components is chosen in this study to achieve higher tensile and flexural strength.

TABLE II PROCESS PARAMETERS

Power of Laser (In Watts)	Scanning Speed (mm/sec)	Thickness (µm)	Hatching Speed (µm)
105	2500	30	50

In addition to providing support structure, the orientation and portion slicing were completed. As a wire EDM allowance, solid supports of height 0.5 mm of the same size substrate plate were provided. All specimens were constructed horizontally. The ASTM E8 sub-size standard was used to design the tensile test specimens. The parts were created using the hexagonal scanning pattern, which is the default parameter used by 3D systems. The components are printed within the building chamber kept in a nitrogen environment to avoid oxidation and any chances of contamination. The entire process took approximately 14 hours to complete the printing. The manufacturing process was followed by removal of completed samples from plate by wire cut EDM.

B. Specimens Preparation

In this experiment specimens were fabricated of size 10×10×5 mm for microstructure investigations. A pin-on-disk wear tester was used to examine the level of wear. As can be seen in Figure 2(a), cylindrical pins with a diameter of 6 mm and overall length of 60 mm were fabricated in a vertical orientation.

Fig 3 (a) Tensile test specimen and (b) Wear test specimens

As shown in Figure 2(b), the bone-shaped tensile test specimen measured 100 mm in length, 24 mm in cross section area, 32 mm in gauge length, and 4 mm in thickness. The wrought specimens were created from commercially available 17-4 PH stainless steel bars.

C. Heat Treatment of Specimens

There are two different cooling rates employed in the heat treatment operations. Specimens underwent the first heat treatment procedure (HT-1), by being heated to 1050°C for 1 hour before being cooled in the furnace. Next, they were aged for 4 hours at 495°C before being cooled in the furnace. The second heat treatment procedure (HT-2) included heating the specimens to 1050°C for 1 hour before allowing them to cool in the air, and then aging them at 495°C for 4 hours before allowing them to cool in the furnace. The recrystallization temperature and typical annealing conditions for 17-4PH stainless steel were taken into account while deciding on the HT conditions. The HT process conditions used in this study are listed in Table 3.

TABLE III HEAT TREATMENT DESCRIPTION

Heat Treatment	Temperature (°C)	Holding Time (Hours)	Cooling medium
HT 1 Homogenization	1050	1	Air
Aging	495	4	Air
HT 2 Homogenization	1050	1	Furnace
Aging	495	4	Furnace

Microstructural investigations were a performed on scanning electron microscope, model JEOL-IT100. Wear behavior was observed on Pin on Disc wear apparatus. Universal testing machines (UTM) model Tinius Olsen HK50 with a maximum loading capacity of 50kN was used for the tensile testing.

III. RESULTS AND DISCUSSIONS

The 17-4 PH stainless steel parts were manufactured successfully using SLM process. The fabricated parts were subjected to microstructural investigations, microhardness, wear testing, tensile and flexural testing.

A. Sem Microstructure Characterization

Analysis of the as-built and heat-treated microstructure of 17-4 PH SLM samples was performed.

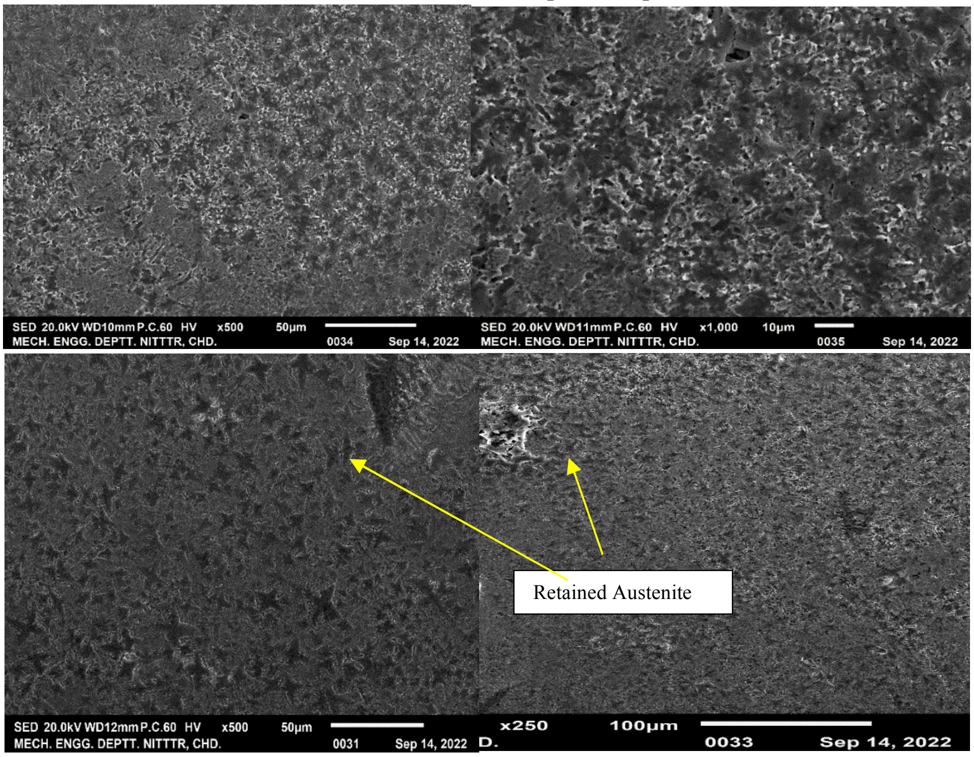

Fig 3 (a) and (b) Microstructure of as built samples. (c) and (d) Presence of retained austenite

Figure 3 (a) and (b) show the microstructure of as-built samples, where the borders of melt pools can be seen. In Fig. 3 (c) and (d), austenite can be seen clustered at the molten pool lines. The samples were made using SLM, therefore, the microstructure changed due to immediate heating and cooling of the selected locations. The continuous heating/cooling cycles modify microstructure in such a manner that the material's reaction to the standard heat treatment method is altered. This is attributed mostly to the production of non-equilibrium microstructures during recurrent heating/cooling cycles [13]. High rate of cooling (10^3°C/s to 10^5°C/s) in the SLM process result in martensite dominance with some preserved austenite in as-fabricated components. Use of an argon environment during the SLM process led to the development of retained austenite, as noted by Rafi. et al. [14]

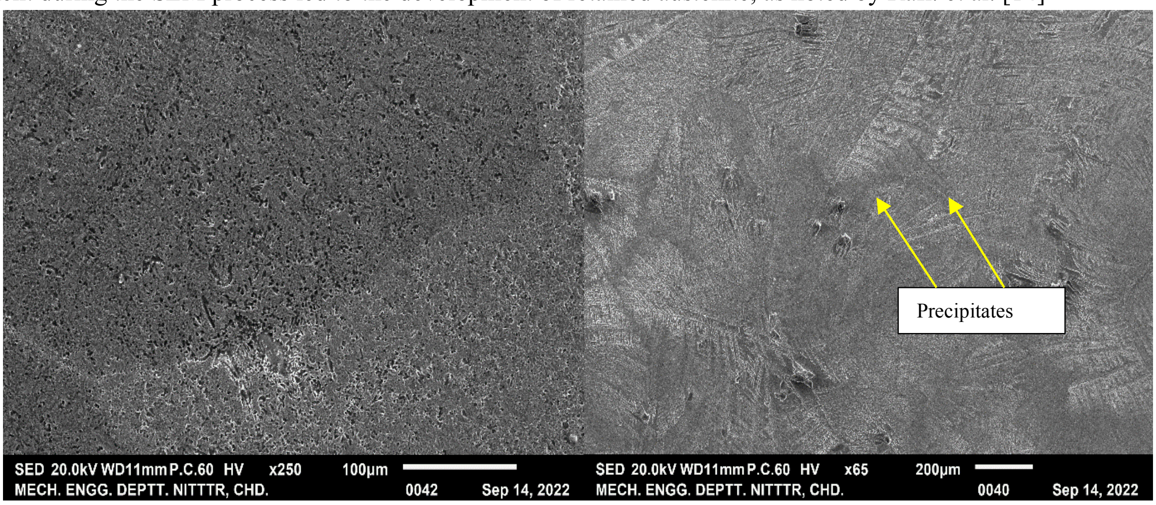

Fig 4 (a) SEM micrographs of HT 1 specimens (b) Precipitates formation

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The microstructure of heat treated samples are show in figure 4(a). The heat treatment homogenized the microstructure with formation of precipitates. The microstructure of individual melt pools exhibited tiny precipitates which were spherical in size and were particularly apparent at dendrite borders as shown in Fig 4(b).

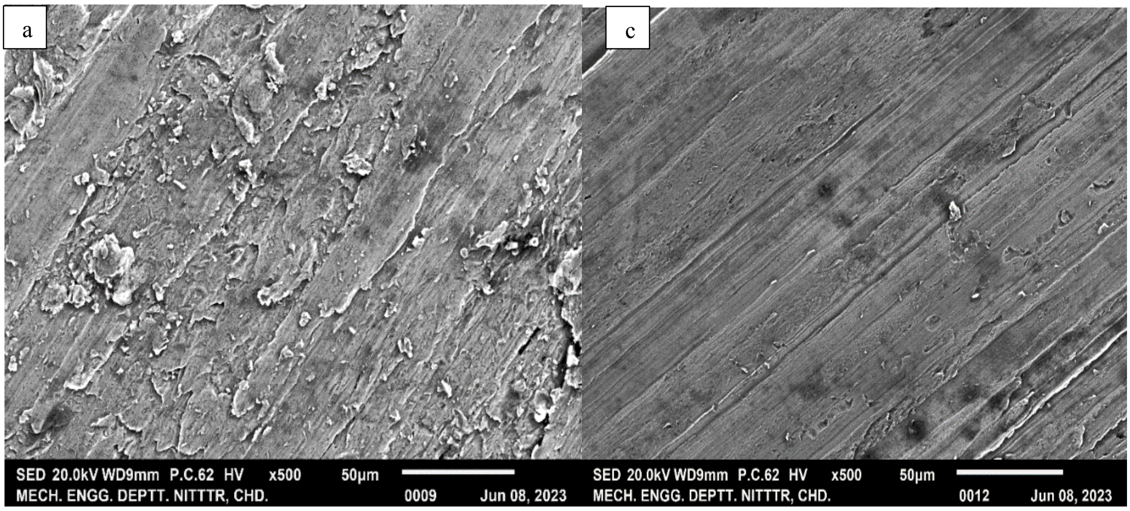
B. Wear Behaviour

The Pin-on-disk wear testing machine was used to conduct the wear test. The value of normal load to the pin against the disk was set to 20N, exerting a controlled normal force and rotational speed of the disk was also set to 500 RPM, ensuring proper contact and sliding conditions. Before testing, the pins were weighed using an analytical balance having a resolution of 0.1mg in order to determine their initial mass. The wear of the pin and the frictional properties were monitored and measured during the test. Based on the measurements, the wear volume or specific wear rate was calculated and are summarized in Table 4.

Table IV Wear rate of specimens

Sample No.	Specific Wear Rate $(mm^3/mN)(\times 10^{-6})$	Mean Specific Wear Rate (mm^3/mN) $(\times 10^{-6})$
AB-A	11.4	11.7
AB-B	12.1	
AB-C	11.7	
HT-1 A	8.2	8.2
HT-1 B	8.0	
HT-1 C	8.5	
HT-2 A	6.4	6.6
HT-2 B	6.9	
HT-2 C	6.5	
Wrought A	9.5	9.9
Wrought B	9.8	
Wrought C	10.4	

The average wear rate for heat treated (HT-2) pins was found out to be lowest with the hardness of 50.9 HV. It is clear that heat treatment in HT 2 yielded the increased pin hardness and is the most wear resistant steel and as built pins with minimum hardness have average specific wear rate of $11.7\times 10^{-6}\, mm^3/mN$.
The images obtained by SEM before the test and worn out surfaces after the test are shown in Figure 5 According to Zum Gahr, the micro-grooving, adhesion, and micro-delamination processes were detected[15]. During micro-delamination, tiny flakes of material are peeled away from the disc's surface. Thus HT 2 pins presented the best specific wear rate of $6.6\times 10^{-6}\, mm^3/mN$.



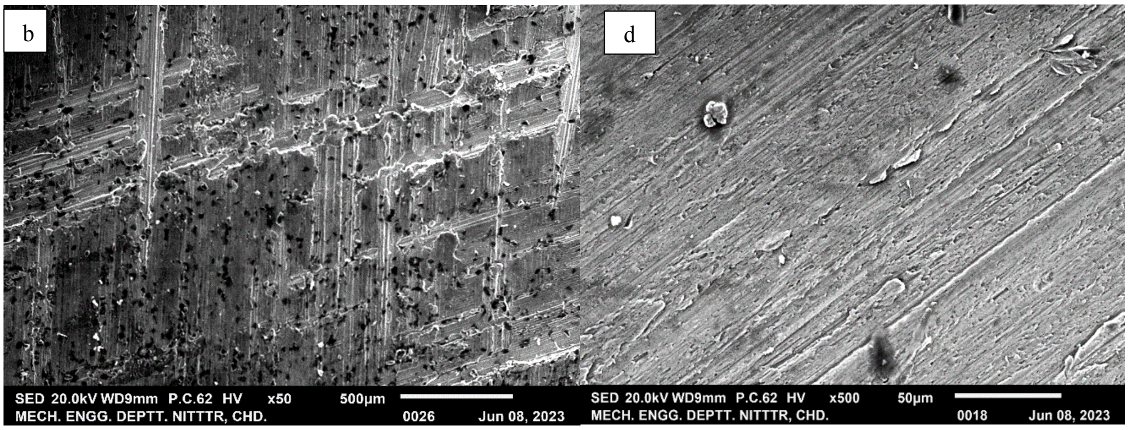


Fig. 6 (a) & (b) SEM images of a pin tip that has been worn down before testing (c) & (d) Scanning electron micrographs of the tested pin's worn tip

C. *Tensile Strength*

The average Tensile Strength of as-built and heat-treated specimens was calculated using the applied load values. The experiment was performed at ambient temperature (24 °C) with a strain rate of 0.001/s.

TABLE V ULTIMATE TENSILE STRENGTH OF TEST SPECIMENS

Test Sample	Serial Number	Applied Load P (kN)	UTS (MPa)	Average UTS
As Built	1	24.6	1020	1022.33
	2	25.5	1058	
	3	23.8	989	
Heat Treated 1	1	26.9	1120	1190.67
	2	27.7	1153.6	
	3	27.4	1142.4	
Heat Treated 2	1	30.50	1276.3	1303.16
	2	32.5	1354.1	
	3	30.70	1279.10	

As-built 17-4PH stainless steel parts were found to have an average ultimate tensile strength of 1022.33 MPa and an average elongation of 14%. Mower et al. (2016) found that the measured tensile strength of additively manufactured components is high as comparable to that of wrought 17-4PH stainless steel. In wrought 17-4PH stainless steel, the ultimate tensile strength was reported to be 1085 MPa. [16]

The average ultimate tensile strength of HT-1 parts produced by SLM was found to be 1190.67 MPa with 17% of elongation. The average ultimate tensile strength of HT-2 parts was observed to be 1303.16 MPa with elongation of 17%. The increase in ultimate tensile strength of HT-2 parts was found out to be 27% when compared with as built specimens. The high tensile strength in HT-2 samples was due to absence of unmelted powder and formation of uniform microstructure.

IV. CONCLUSIONS

Various metallic designs were successfully built utilizing 17-4PH Stainless Steel powder and selective laser melting technology. Microstructural analysis, wear behavior and tensile testing were performed on the additively built, heat treated and wrought specimens. The produced pieces had a smooth surface morphology with no imperfections.

1) The HT 2 specimens showed best wear resistance with and average specific wear rate of 6.6 × $10^{-6}\ mm^3\ /mN$ and HT 1 showed lower wear resistance of 8.2 × $10^{-6}\ mm^3\ /mN$ followed by as built and wrought samples with wear resistance of 11.7 × $10^{-6}\ mm^3\ /mN$ and 9.9 × $10^{-6}\ mm^3\ /mN$. respectively.

2) The results showed that UTS of SLM HT-2 specimens was higher when compared to the as built specimens.
3) The SLM 17-4 PH SS's mechanical characteristics were altered as a result of the precipitation hardening and microstructure homogenizing impacts of heat treatment. Ultimate tensile strengths and wear behavior were improved by heat treatment. It was discovered that the heat treated samples exhibited the high ductility than the as-built samples.

V. ACKNOWLEDGEMENT

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