MICROSTRUCTURE AND MICROHARDNESS OF HEAT TREATED 17-4 PH STAINLESS STEEL PRODUCED BY LENS TECHNIQUE

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The current study focused on optimizing heat treatment parameters of 17-4 PH stainless steel produced on a LENS platform. The fundamental objective of the current study as to attain the microhardness of the as received machined sample which was ± 400 HV. This was achieved by firstly printing 17-4 PH stainless steel coupons using LENS system. Subsequently, the coupons were subjected to various heat treatments which includes solution treatment at 1100℃ for 2hrs followed by furnace, air and water cooling. Furthermore, the aging temperature was varied from 400℃ to 510℃, while the aging time was varied from 10 minutes to 10 hours. Material characterization (microstructural evolution and microhardness) of as-built and heat treated samples was conducted. The microstructure in as-built condition was inhomogeneous and became homogeneous after homogenization heat treatment. Moreover, the optimum aging temperature and time, which produced a microhardness of 400 HV, are 400 ℃ and 30 minutes respectively.

INTRODUCTION

Additive manufacturing (AM) also known as 3D printing is a relatively new manufacturing method, which has attracted industries such as aerospace, chemical and biomedical. The mechanism of 3D printing involves manufacturing an object from a 3D computer aided design (CAD) model by building layer upon layer until a complete 3D part is produced [9]. The attraction of 3D printing is because it has the ability of producing parts with superior mechanical properties as opposed to traditional manufacturing methods [1]. Laser Engineered Net Shaping (LENS) is one of the additive manufacturing techniques, and this technique manufactures a part by simultaneously depositing the powder metal and irradiating laser beam on powder particles deposited [8]. The 17-4 precipitation hardened stainless steel is one of the most used material in industries such as aerospace, chemical and biomedical. This is attributed to the fact that 17-4 PH stainless steel possesses outstanding mechanical properties and corrosion resistance [2]. However, the dynamics of AM inherently result in an inhomogeneous microstructure, which can results in detrimental effects of the printed part[10]. This suggest that parts produced by LENS technique should be subjected to homogenizing heat treatment.

Gu *et al*[4] printed 17-4 PH stainless coupons using a direct metal laser sintering platform, and they reported that they observed a microstructure with 70 % proportion of martensitic phase and 30 % of austenitic phase. High proportionality of austenite was caused by the fact that processing was conducted in a nitrogen environment, which is known as an austenite stabilizer. Meanwhile, Cheruvathur *et al* [3], also printed 17-4 PH stainless steel components in a nitrogen environment, and the as-printed microstructure showed 50 % martensite and 80 % austenite. However, after subjecting the as-printed components to homogenizing heat treatment, the proportion of austenite decreased to below 10 %. The aforementioned findings by Cheruvathur *et al* [3], differs with Gratton's [5] findings, which argued that the microstructure did not change when the printed components were subject to solution heat treatment. Concerning microhardness, Nezhadfar *et al* [5] investigated the effect of shielding gas on the microhardness of laser-powder

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The author is enrolled for PhD in the Department of Mechanical Engineering Sciences, University of Johannesburg, Gauteng, South Africa bed fusion (L-PBF) 17-4 PH stainless steel component. They established that the component produced in nitrogen environment had higher hardness as opposed to component produced under argon environment and this is because the thermal conductivity of argon is 40 % less than that of nitrogen. Moreover, Nezhadfar *et al* [6], observed that heat treating 17-4 PH stainless steel in argon environment resulted in higher hardness as opposed to heat treating in nitrogen environment. Additionally, Ponnusamy *et al* [7], argued that printing 17-4 PH stainless steel in vertically orientation, resulted in higher hardness than printing in horizontal and diagonal orientation.

This study will seek to attain a microhardness of around 400 HV of as-received machined 17-4 PH stainless steel, which is said to have been aged at 510℃ for 4 hours. However, when the same aging parameters were employed to a 17-4 PH stainless steel produced by LENS platform, the hardness was found to be extremely higher(470 HV) than that of as-received machined 17-4 PH stainless steel. Therefore, the current study investigated the effect of heat treatment parameters (time, and temperature) on 17-4 PH stainless steel produced by LENS technique.

METHODOLOGY

The current study used TLS 17-4 PH stainless steel powder with spherical particle size ranging from 40 to 90 µm, which are shown in **Figure 1**. The energy-dispersive X-ray spectroscopy (EDS) elemental composition of 17-4 pH used in the current study is shown in **Table 1**. The printing of coupons were conducted on a LENS Optomec 850-R platform with a laser power, scanning speed, powder feed rate and layer thickness of 300 W, 12.7 mm/s, 4.5 rpm and 0.06 mm respectively. Subsequently printed coupons were subjected to heat treatment at various time and this can be seen in **Table 2**. Material Characterization (microstructural evolution and microhardness) of as-built and heat treated coupons was conducted. The coupons were sectioned, mounted, grinded, polished and the etched in FRY'S reagent. The evaluation of microstructure was carried out on optical microscope (Olympus BX51M) and Scanning electron microscope (Joel JSM-6010PLUS/LA). Microvickes hardness tester (Zwick/Roell) was used to measure the hardness of as-build and heat treated samples. Furthermore, the samples were subjected to a load of 300 gf for 10 seconds,

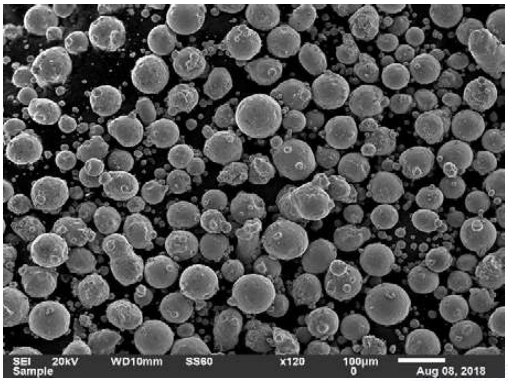

Figure 1: 17-4 PH stainless steel powder particles

Table 1: major elementary composition of 17-4 PH stainless steel

Fe	Cr	Ni	CU	Si
74.35 %wt	**17.55 %wt**	**3.83 %wt**	**3.61 %wt**	**0.65 %wt**

Table 1: heat treatment parameters

Treatment	Temperature(˚C)	Time	Cooling method
Homogenizing	1100	2 hrs	Furnace, air, water
Aging	400-510	10 min- 10hrs	air

RESULTS

The microstructure in an as-built condition is characterized largely by martensitic phase with some dendritic structure, which formed because of rapid cooling associated with additive manufacturing as depicted in **Figure 2A**. The as-built microstructure further shows some melt pool boundaries that formed due to re-melting and some porosity that formed during processing, which is probably due to hatch spacing. **Figure 2B** shows a microstructure after homogenization treatment and it be seen that the microstructure is homogenous, with melt pool disappearing.

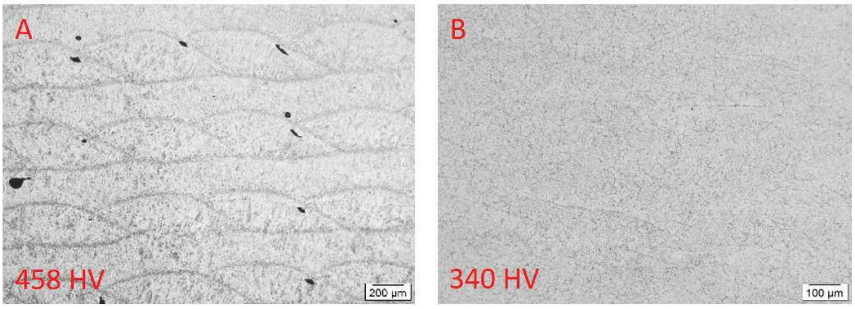

Figure 2: OM images A. as-built microstructure, B. homogenized microstructure

Figure 3 shows SEM microstructures of various cooling methods after homogenization treatment at 1100°C for 2hrs. **Figure 3A**, **3B** and **3C** shows a microstructure after furnace, air, and water cooling respectively. It was found that furnace cooling resulted in the formation of grains and small white Niobium carbides precipitates whereas for both air and water cooling, no grains formation was observed as well as and Niobium carbides precipitates.

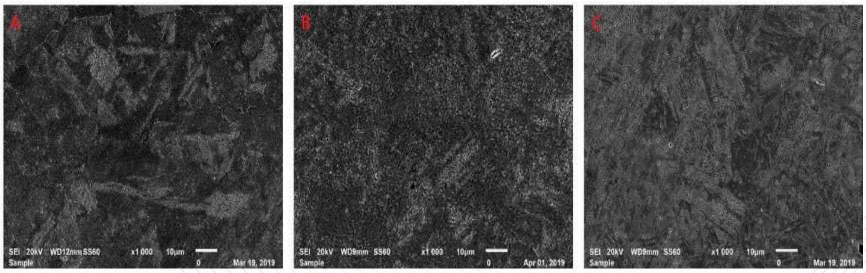

Figure 3 SEM micrograph Homogenized at 1100℃ for 2hrs, A. furnace cooled, B. air cooled, C. water quenched.

These Niobium precipitate can be seen clearly after aging treatment, where they have fully developed and this is shown in **Figure 6**. The fact that grains formed during furnace cooling is attributed to slow cooling that is associated with furnace cooling and grains did not form during water and air cooling because of rapid cooling. The formation of niobium carbides is driven by slow cooling, which allows them to diffuse out of solution and form small white phases.

Additionally there is a correlation between the hardness results in **Figure 4** and the microstructures in **Figure 3**. It can been seen that furnace cooling produced a microhardness value which is higher than water and air and cooling regardless of large grains that formed during furnace cooling as depicted in **Figure 3A**. This suggest that the presence of Niobium carbides precipitates after furnace cooling provided the resistance to plastic deformation more than water and air cooled during hardness testing and this also implies that the effect of niobium precipitates on hardness supersede the effect of grainsize.

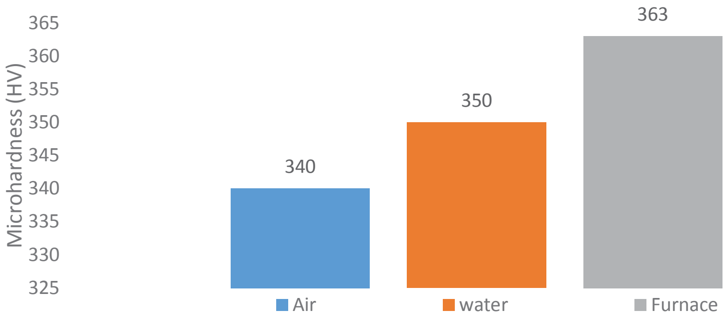

Figure 4: Microhardness as function of various cooling methods

Subsequent to homogenization treatment, several aging treatment at 500 ℃ were conducted as shown in **Figure 5**. **Figure 5** shows that hardness increased with increment of aging time from 10 minutes and reach a peak at 1 hour and this is due to the increase of density of Niobium carbides precipitates. However, the hardness started to decrease when the aging time increased from 1 hour to 4 hours and this might be because, Niobium precipitates coarsen with time and become incoherent with the matrix, therefore not effective in hardening. Furthermore, it can be seen that the hardness further increased when the aging time increased from 4 hours to 10 hours. This phenomenon has not been reported in an open literature and requires an in depth analysis of what might be causing the hardness to increase again.

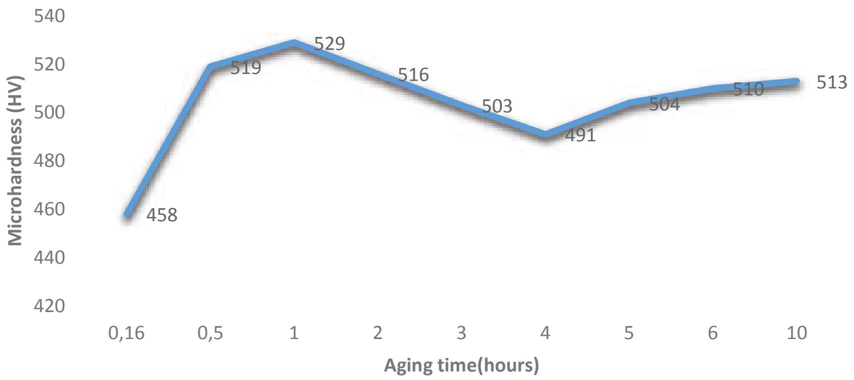

Figure 5: Microhardness as a function of aging time

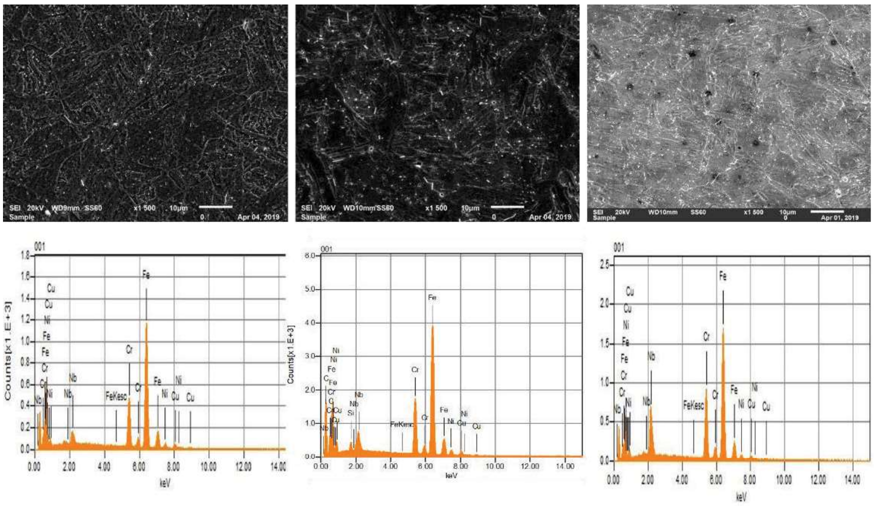

Figure 6: Depicting SEM images and their EDS at 500℃ and various aging time A. 10 min, B. 30 min C. 1 hour.

The hardness required (±400) is not attained by varying the aging time and keeping the temperature constant at 500 °C. **Figure 7** shows a graph of microhardness as a function of aging temperature and the aging time was kept constant at 30 minutes. The graph shows that the microhardness decreased due to decrease of aging temperature and the optimum temperature is found to be 420℃. This suggest that Niobium precipitates are sensitive to heat than the aging time because the hardness decreased significantly due to variation of temperature as opposed to variation of time.

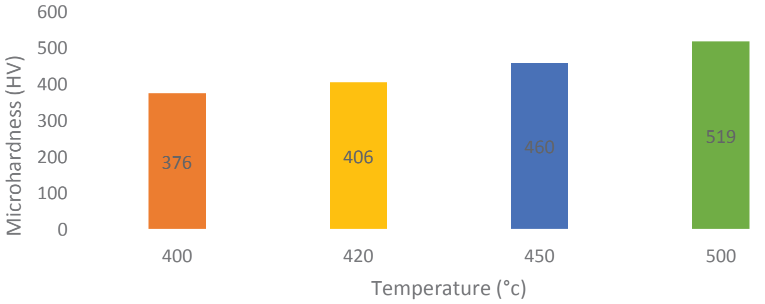

Figure 7: Microhardness as a function of temperature

CONCLUSION

The current study deduced that homogenizing heat treating at 1100℃ for 2 hours was effective in forming a homogenous microstructure. It was further established that the furnace cooling enables the formation of Niobium precipitates, which resulted in higher hardness as opposed to water and air cooling. Moreover, the current study concluded that the formation of Niobium precipitates is more sensitive to heat (aging temperature) than the aging time and the optimum time and temperature, which produced the required microhardness, is 30 minutes at 420℃ respectively.

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