Investigation of the effect of alkaline environment on mechanical properties of additively manufactured 17-4 PH Stainless Steel parts produced using recycled metal powder

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Abstract

Metal additive manufacturing (AM) has shown its advantages over conventional manufacturing methods in producing complex components. Re-usage of metal powder after completion of the AM process for the next batch of the manufacturing cycle is one of the ways to make AM more cost-effective and environmentally friendly. 17-4 precipitation hardening stainless steel (17-4 PH) offers corrosion resistance, high strength, and toughness making it a widely used AM material. Recycling of metal powder in AM has inherent impacts on materials' performances. Such practice makes it imperative to fully comprehend the performances of AM parts produced by re-used metal powder. In the current research, the authors have performed an investigation of the effect of alkaline environment on the mechanical properties of additively manufactured 17-4 PH parts produced using recycled metal powder. This study is conducted due to the lack of scientific knowledge in this area. The authors performed immersion testing of recycled powder produced 17-4 PH parts in aqueous alkaline solution and subsequently conducted weight loss analysis, tensile testing, hardness testing and microstructural analysis. Results of mechanical properties were also compared with those of the as-built recycled and virgin powder produced 17-4 PH AM parts. The findings of the present study indicated changes in the weight, tensile strength, ductility, hardness, and morphology of the recycled AM parts after subjected to alkaline environment. These changes in material performance and morphology could be owing to the reaction to the corrosive effects of active hypochlorite ion (OCl-) present in alkaline solution.

Keywords: additive manufacturing, laser powder bed fusion, 17-4 PH stainless steel, recycled metal powder, alkaline environment, microstructure

1. Introduction

AM is a promising method for producing complex components with improved design in a single unit, which is produced in several subparts by conventional manufacturing methods and assembled as a final product. Therefore, AM provides many advantages, including shorter time to market, mass reduction with better structural integrity whilst allowing optimization of complex designs that are otherwise impossible to build, lower energy usage, and overall lower cost of production due to less manpower involvement and material wastage. It can support a broad range of applications ranging from medical to aerospace (Lyons et al., 2021), (Katsarelis et al., 2019; Blakey-Milner et al., 2021). According to the ASTM-F42 committee guidelines, AM processes can be classified into seven categories, i.e. vat photopolymerization (VP), material jetting (MJ), binder jetting (BJ), material extrusion (ME), sheet lamination (SL), powder bed fusion (PBF), and directed energy deposition (DED) (Srivastava et al., 2022). The PBF process is an advanced manufacturing technology that involves layer-by-layer addition of material to fabricate actual parts using a 3-dimensional solid computer-aided design (CAD) model. The heat energy source used in the PBF process for the irradiation, fusion, and melting of powder particles is either laser or electron beam (Singh et al., 2021). Laser powder bed fusion (LPBF) is the most commonly used metal AM technique (Santecchia et al., 2020).

Using recycled metal powder in the LPBF technique provides significant cost and environmental benefits; however, it could have adverse effects on the mechanical performance of the manufactured parts. Several studies have focused on examining the use of recycled metal powder in LPBF production of metal components (Gorji et al., 2020; Soltani-Tehrani et al., 2023; Lanzutti et al., 2023; Giganto et al., 2022; Santecchia et al., 2020). It has been found that the iterations of the recycling process bring about changes in the physical and chemical properties of virgin powder particles which consequently affect the properties of manufactured products (He et al., 2022). Gorji et al. (Gorji et al., 2020) studied pore formation in recycled powder particles and observed a broader pore size distribution along with a higher pore density in recycled powders than those determined when using virgin particles. Their findings suggested lower hardness and higher effective moduli for the recycled powder particles. Terrassa et al. (Terrassa et al., 2018) studied the influence of reusing powder on particle morphology, flowability, agglomeration, chemical composition, and

microstructure. Their study suggested that powder and deposition quality can be maintained with controlled processing through ten powder reuse cycles with minimal processing between cycles.

17-4 PH offers a good combination of corrosion resistance, high strength, and toughness (Cheruvathur et al., 2016; Sabooni et al., 2021) making it suitable for many industrial applications. The manufacturing process alone can have a significant influence on the microstructure of fabricated 17-4 PH parts. They constitute a fully martensitic structure in the wrought form (when produced by conventional methods) which is often followed by heat treatment for strengthening by the precipitation of highly dispersed nanosized Cu-rich particles (Sun et al., 2018; Cheruvathur et al., 2016). Whereas, AM produced 17-4 PH parts are known to possess a non-equilibrium microstructure (Sun et al., 2018) owing to the rapid cooling rate and the building orientation of parts, i.e. horizontal or vertical to the build platform (Yadollahi et al., 2017; Mahmoudi et al., 2017). The as-built 17–4 PH AM parts have martensitic structure with a fraction of retained austenite phase (LeBrun et al., 2015). Depending upon the gas atmosphere in the LBPF manufacturing process, 17-4 PH mainly has martensitic structure with small amounts of retained austenite under argon and from 50% to 75% of the austenite phase (depending on the component orientation) with the remaining martensite under nitrogen (Sabooni et al., 2021; Rafi et al., 2014). Cheruvathur et al. (Cheruvathur et al., 2016) research compared different heat treatment processes for as-built 17-4 PH produced by AM to achieve uniform microstructure. Their study proved that homogenization annealing at 1150 °C helped breaking the dendritic solidification structure leading to approximately 90% martensite and 10% retained austenite; however, still 100% martensite microstructure could not be achieved. Hence, the variation in microstructure due to the range of processing parameters in AM methods and post-built heat treatment strongly influences the mechanical performance of AM parts.

The mechanical performance of 17-4 PH stainless steel produced by LPBF has been investigated in the past both for parts produced by virgin metal powder (Weaver et al., 2021; KALITA and JAYAGANTHAN, 2023; Sripada et al., 2022; Yadollahi et al., 2020) and recycled metal powder because of the associated cost benefits of re-used metal powder (Giganto et al., 2022; Soltani-Tehrani et al., 2020; Ahmed et al., 2020). Subsequently, the corrosion of stainless steel, especially 17-4 PH, in aqueous environments has been studied by other researchers (Dartar Öztan et al., 2002; Martínez-Aparicio et al., 2023; Tavakoli Shoushtari, 2010; Xuefei Wang et al., 2023). However, there is a gap in the study of the effect of alkaline

solutions on the mechanical performance of recycled metal powder-produced AM parts. Therefore, this study aimed to fill this gap. This study involved the following four stages: (1) producing specimens from recycled metal powder using the LBPF process; (2) conducting immersion test of the as-built specimens (without post-built heat treatment) in alkaline solution for specified durations; (3) conducting tensile and hardness testing of specimens; and finally, (4) microscopic characterization of fractured surface of specimens from tensile testing to further understand the effects of alkaline solution on the material's morphology.

2. Experimental Details

2.1 Materials

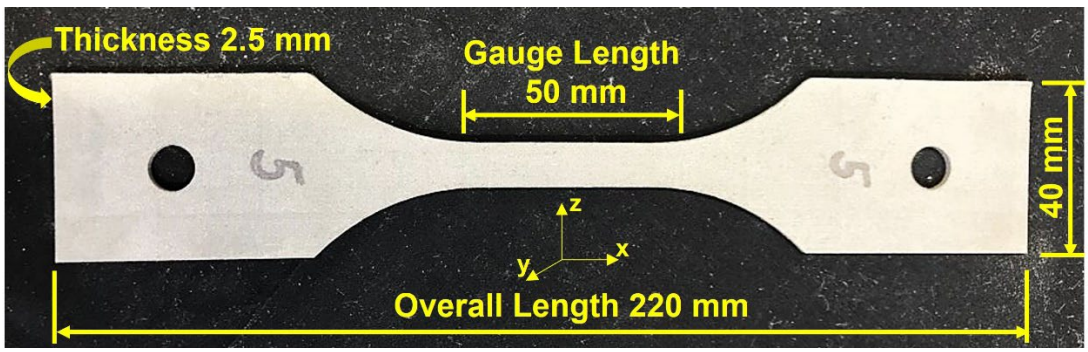

Figure 1: Typical as-built 17-4 PH specimen produced by LPBF technique

Flat tensile test specimens of 17-4 PH stainless steel (also known as 630 steel according to the AISI standard) were additively manufactured using ProX DMP 300 3D metal printer (3D Systems, Inc., USA). The dimensions of the specimens are as shown in Figure 1. All specimens were printed (horizontally i.e., parallel to the build platform) using selective laser melting (SLM) technology, which belongs to the family of LPBF processes. The steps followed for specimen production are as follows.

First, a 3D solid CAD model file was processed using ProX DMP manufacturing CAD/CAM software, and then the manufacturing file (FAB2 or FAB3) was created. The SLM Machine was prepared, the FAB2 or FAB3 file was uploaded onto the machine, and the layering cycle was started where a very high-precision laser is used to melt the powder particles and build the required geometry. With every completed cycle, the build platform was lowered in the vertical z-direction respective to the specimen orientation, as shown in Figure 1, and a fresh layer of metal powder was evenly spread across the platform with the help of a coater blade extract metal powder from the reservoir in the chamber. These cycles were repeated until the

specimens were fabricated. During the manufacturing process, the metal powder was kept within the 3D printer under an inert, dry nitrogen atmosphere. The manufacturing parameters used in the 3D printing process are listed in Table 1.

The amount of energy delivered per unit volume of the powder bed, known as the Volumetric Energy Density (VED) was calculated using Eq. 1 (Giganto et al., 2022) and was found to be equal to 62.5 J/mm^3.

$$VED\ [J/mm^3] = \frac{\mathrm{P\ [W]}}{\mathrm{v[mm/s]\cdot h[mm]\cdot L_t[mm]}} \quad (1)$$

Table 1: Manufacturing parameters used in LPBF process in the current study

Parameters	Value	Unit
Laser power, P	150	W
Scan speed, v	1200	mm/s
Layer thickness, L_t	40	µm
Hatch distance, h	50	µm
Inert gas atmosphere	Nitrogen	-
Scan pattern	Hexagonal cells	-
Cell size	25	mm
Cells overlap	180	µm

The metal powder used for producing specimens is LaserForm 17-4 PH (B) (3D Systems, Inc., USA). The chemical composition of virgin 17-4 PH metal powder is mentioned in Table 2. In the current study, 17-4 PH metal powder which had gone through eight iteration cycles of LPBF process in ProX DMP 300 3D metal printer prior to the current study, was used. The standard procedure for recycling metal powder in the 3D printing laboratory involves recovering unused metal powder from 3D printer using a specialized vacuum cleaner and transferring the powder to the recycling unit where the powder is run through a 70 µm sieve screen for separating the bigger powder particles which fused together during the manufacturing process in the chamber. The filtered metal powder (particle size <70 µm) is then pre-conditioned by baking at 80 °C for 16 hours in a drying oven and loaded back into the 3D printer for re-use.

Table 2: Chemical composition of virgin 17-4 PH metal powder (3DSystems)

Elements	Cr	Ni	Cu	Mn	Si	Nb	Fe
Wt. %	15 - 17.5	3 - 5	3 - 5	< 1	< 1	0.15 - 0.45	Bal

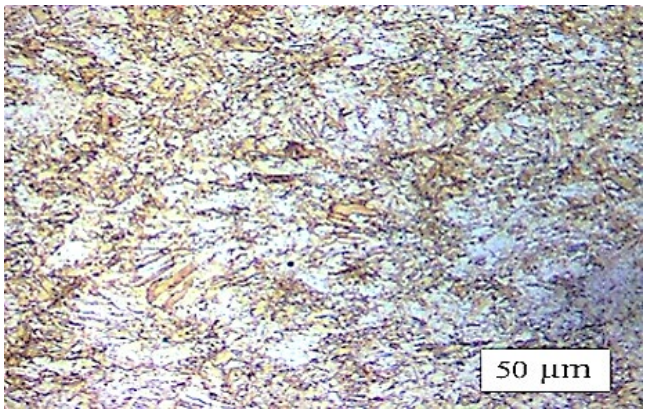

Figure 2: Surface morphology image (10x) of the as-built specimen taken in z-x plane prior to immersion and mechanical testing

Figure 2 is an optical micrograph (taken at 10x magnification using Olympus BX61 optical microscope) showing the surface morphology of the as-built 17-4 PH specimen. Prior to imaging, the specimen was cut and placed in a mold filled with epoxy resin and hardener liquid with a ratio of 70:30. The specimen surface was then ground using rough to lower grit's silicate carbide papers of 400 µm to 2500 µm on a rotating disc before diamond polished, and finally etched using nitric acid.

The surface roughness of as-built specimens was also measured within the gauge length region (see Figure 1) using Mitutoyo Surface Profilometer (SV- C3200) following the measurement conditions as specified in Table 3. The average roughness (Ra) and root-mean-square roughness (Rq) of 6.51 µm and 8.11 µm, respectively were noted. These roughness values are more and less in the same order of the reported roughness of 10-30 µm in the published literatures. Generally, SLM-produced parts tend to have higher roughness than 1-2 µm typical Ra of parts fabricated using milling process (Di Wang et al., 2016; Sander et al., 2018).

Table 3: Surface roughness measurement conditions

Instrument	SV- C3200
Measurement Length	2.4 mm
Measurement Pitch	0.0005 mm
Measuring Speed	1.00 mm/sec
Z1-axis Range	80.0 µm
Sampling Method	X-axis Pitch
Z Gain Adj. Ratio	1.045274

2.2 Methodology

2.2.1 Immersion Testing

Immersion testing of as-built 17-4 PH stainless steel specimens was conducted in alkali solution at room temperature under open-air condition. Nine as-built specimens were immersed for different time periods i.e. 1, 3 and 5 days, with three specimens experimented at each duration and referred to D1, D3 and D5, respectively. Household bleach containing up to 5% sodium hypochlorite (NaOCl) was used as it contains chloride ions which are aggressive to metals. The details of the chemical composition of the aqueous solution used are stated in Table 4. PH level of the solution was checked prior to immersing specimens using universal PH indicator strips. It was found to be 13, suggesting that the solution was highly alkaline. All specimens were weighed before and after the immersion test to measure any weight loss.

Table 4: Chemical composition of the aqueous solution

Name	EC No.	CAS – No.	Content
Sodium Hypochlorite Solution (NaOCl)	231-668-3	7681-52-9	1-5%
C12-18 alkyl dimethylamine oxide	-	68955-55-5	1-5%
Sodium Hydroxide	215-185-5	1310-73-2	<1%

2.2.2 Mechanical Testing

Weight loss measurements, tensile and hardness testing were performed on all nine specimens (D1, D3 and D5) post-immersion, and on all the as-built specimens (referred to D0) for benchmarking purpose. Tensile testing was carried out using a Universal Tensile Tester at a very low strain rate of 1 mm/min and 50 kN of pre-load at room temperature. Each tensile test was repeated at least three times for better repeatability. Ultimate Tensile Strength (UTS) and the percentage elongation (%EL) of the material were considered for analysis. Hardness testing was also conducted using Rockwell Hardness Tester (HRA). The hardness test force applied was 60 kg-f (588.4 N) and testing was carried out at room temperature by making four indentations on the surface of specimens. The average of four hardness measurements was taken for analysis purpose.

2.2.3 Microstructural Analysis

The microstructural analysis of the fractured cross-sections of the tensile tested specimens was conducted using the Scanning Electron Microscopy (SEM) Zeiss, EVO MA10. For the analysis, a small segment near

the fractured region was carefully cut from the representative specimens of each test condition (D0, D1, D3 and D5).

3. Results & Discussion

3.1 Weight Loss Analysis

The weight loss measurements (shown in Figure 3) suggested that post-immersion testing, all specimens irrespective of test duration, have undergone weight loss albeit the loss is less than 1% in all cases. Moreover, it was observed that immersing the specimens for 5 days led to higher weight loss which can be seen in case of D5 specimens, except for D5's specimen 3.

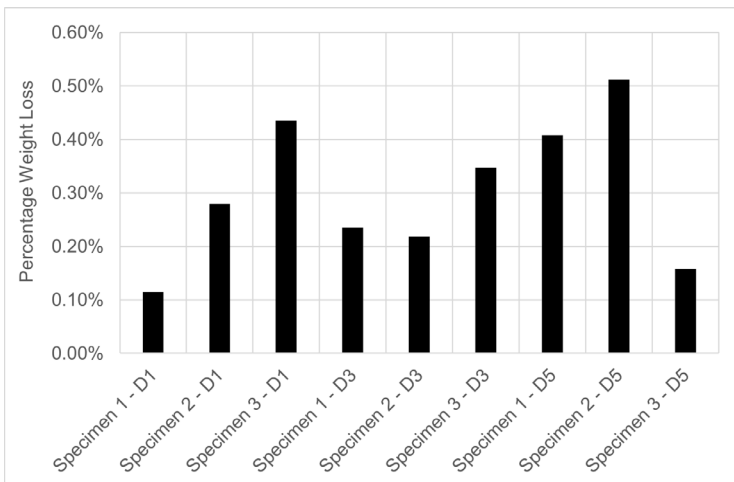

Figure 3: Percentage weight loss of the as-built specimens post-immersion testing

3.2 Tensile Testing

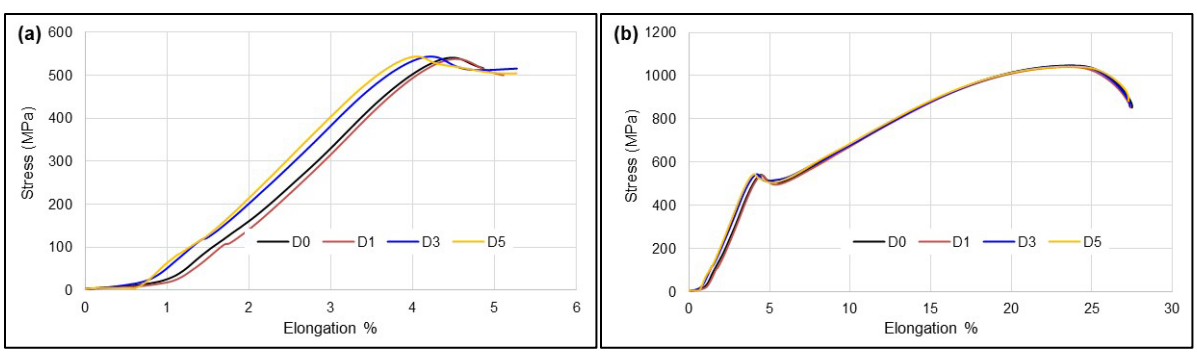

Figure 4: Stress vs. Elongation curves: (a) up to 5% EL and (b) until failure

Figure 4 compares the typical representative tensile deformation behavior of specimens, one from each category, D0, D1, D3 and D5. Figure 4(a) shows the change in stiffness (slope of curve within the elastic or linear region) of specimens. Stiffness can be seen to increase in case of D5 (the most increase), followed by D3, relative to D0. Whereas, in case of D1, stiffness has dropped a little but remained close to D0 indicating no significant change in mechanical behavior after only one day of immersion in aqueous solution. The overall performance of 17-4 PH stainless steel specimens under tensile loading until failure of specimens is demonstrated in Figure 4(b). At first, it appears that the tensile behavior has not been affected beyond elastic region as there is an overlapping of the stress-elongation curve in all cases. However, Figure 5 shows further analysis of tensile testing data and reveals the change in UTS and EL due to the exposure to aqueous environment. It can be seen in Figure 5 that the average UTS reduced by approximately 11 MPa in the case of D1 and around 6 MPa in the case of D3 and D5, relative to UTS of D0. Similarly, the average EL (%) also decreased in case of D1, D3 and D5 compared to D0, however the change observed is very marginal. Furthermore, UTS and %EL for as-built 17-4PH specimens made using virgin metal powder are 1100 ± 50 MPa and 16 ± 2 %, respectively, as stated by manufacturer (3D Systems, Inc., USA) in material datasheet (3DSystems). When comparing the virgin and the as-built recycled powder AM (D0) performances, as shown in Figure 5, there was an UTS dropped by 50 MPa and increased elongation by approx. 11 % owing to the use of recycled metal powder through the LBPF process cycles. Such an increase in material ductility after metal powder recycling could be attributed to higher flowability of metal powder due to lower cohesiveness in metal powder particles after recycling (Nezhadfar et al., 2018).

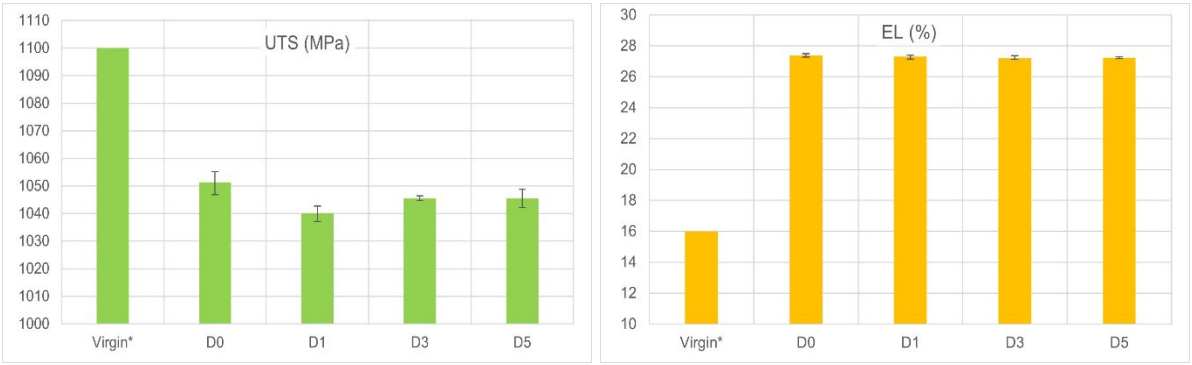

Figure 5: Tensile test results: Ultimate Tensile Strength (UTS, Mpa) and Elongation (%EL). *Data source for as-built specimen made of virgin 17-4 PH metal powder (3DSystems)

3.3 Hardness Testing

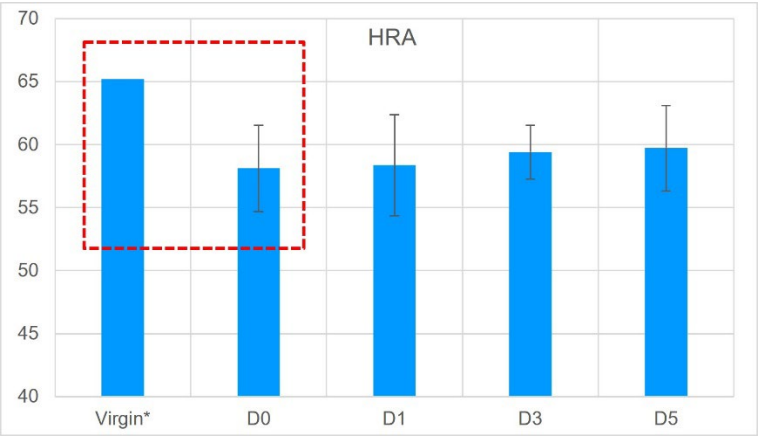

Figure 6: Hardness test results of 17-4 PH parts produced by recycled metal powder. *Data source for as-built specimen made of virgin 17-4 PH metal powder (3DSystems)

Using Rockwell hardness testing machine in A scale, the HRA (hardness) values of 17-4 PH specimens subjected to the aqueous environment (D1, D3 and D5) and as-built specimens (D0) were measured. Figure *6* shows the average HRA values taken after four indentations on three specimens each for the case of D0, D1, D3 and D5. As-built specimens (D0) showed the lowest average HRA value of 58.12 whilst the highest average HRA value of 59.73 was noted for the case of D5 specimens, therefore, an increase of 2.7 % in hardness was observed. Overall, an upward trend in the hardness of recycled 17-4PH can be seen with an increase in duration of specimens' immersion in the alkali solution. In addition, the manufacturer's specified hardness of the as-built 17-4 PH specimens made from virgin metal powder is 300 ± 20 HV5 (equivalent to 65.2 HRA) (3DSystems). When comparing the hardness values of the recycled D0 (58.12 HRA) and of the virgin metal powder specimen (65.2 HRA) as shown in Figure *6* (in red dotted box), a reduction of 10.9 % in hardness of 17-4 PH parts after re-using the metal powder in LBPF process through eight iterations cycles was determined. These findings align with the percentage elongation results shown in Figure *5*.

3.4 Microstructural Analysis

Figure *7* shows the SEM images of fractured surfaces of specimens post-tensile testing for each case of D0, D1, D3 and D5, taken at two different magnifications (250x and 1000x). A detailed study of microstructural defects shows dimples (or porosities) on the fractured surface of tensile test specimens in all cases. Other researchers have reported similar dimples formation phenomenon, which are attributed to lack-of-fusion, trapping of gas, and inclusions (un-melted powder and spatter particles), that cause the

nucleation of voids until they coalesce during the AM process (Giganto et al., 2022). In the current study, higher density of deeper dimples was observed on the fractured surface of D5 specimen compared to the fractured surfaces of D0, D1 and D3 indicating changes in material morphology in D5 due to longer exposure to aqueous solution. These SEM observations (Figure 7) demonstrate the evolution in the change of material morphology from D0 to D5.

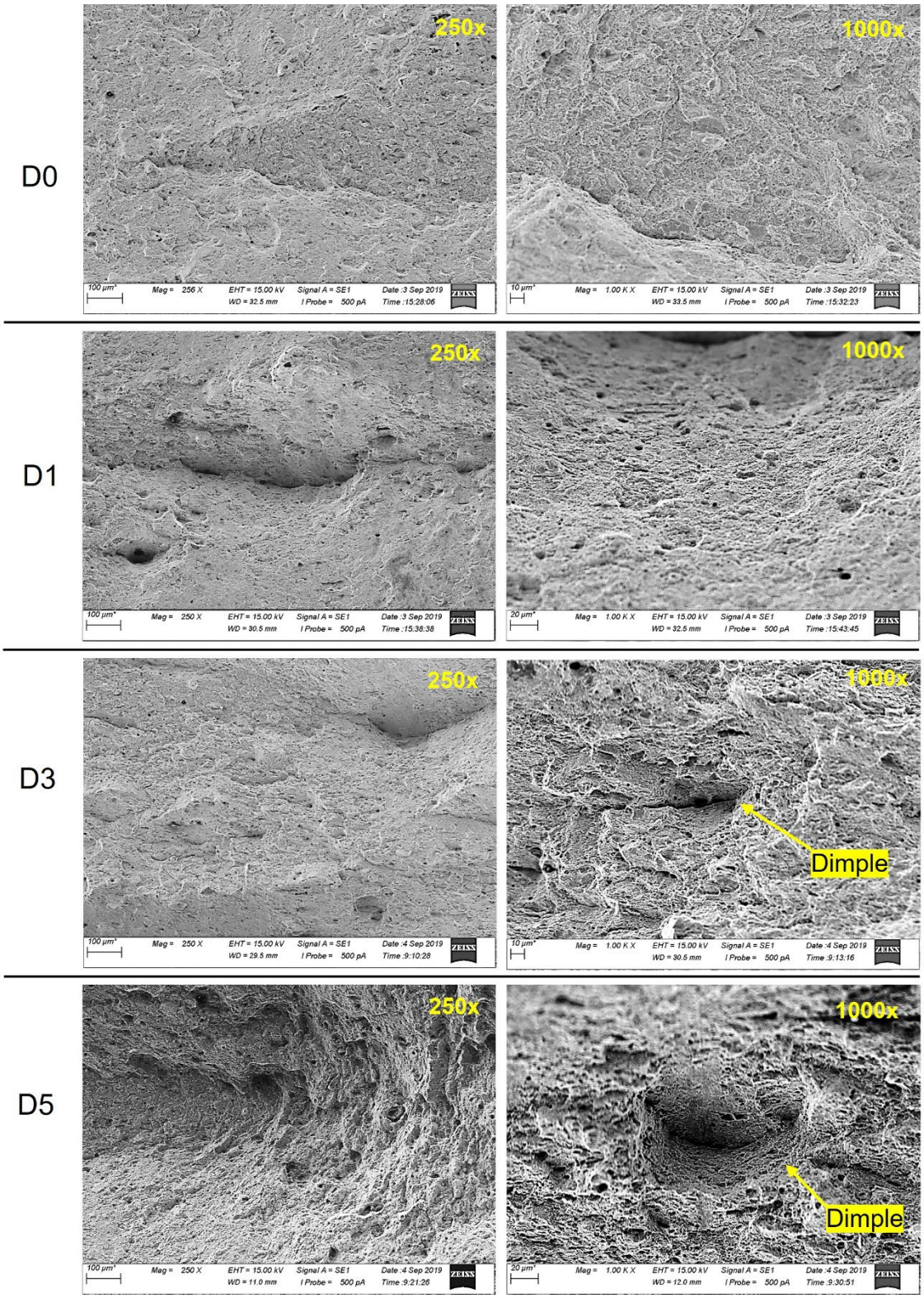

Figure 7: SEM images of the fractured surfaces of tensile test specimens from D0, D1, D3 and D5 taken at 250x and 1000x showing changes in material morphology

4. Discussion

In the present study, the effect of aqueous solution on the mechanical performances of the recycled 17-4PH metal powder AM parts is investigated. The weight loss analysis indicates the aggressive action of aqueous solution on 17-4 PH. Also, longer exposure leads to higher weight losses. This phenomenon could be correlated to the presence of hypochlorite ions (OCl^-) in aqueous solution. Hypochlorites are alkaline oxidizing agent and known for their corrosive nature (Gaur et al., 1994). It is well known that the free corrosion potential of stainless steels is ennobled by the presence of oxidizing species thereby increasing the risk of localized corrosion and transpassive dissolution (Moayed and Golestanipour, 2005). Furthermore, tensile testing results clearly revealed that recycling metal powder has strong influence on the tensile behaviour of 17-4 PH AM parts, where parts loss their higher load carrying capacity and become more stretchable (indicating increased ductility). The exposure to aqueous solution degrades the tensile strength of the material, along with some noticeable effects on the stiffness and elastic deformation (Figure 4a). There is a negligible effect on plastic deformation of material (Figure 4b). In addition, hardness testing findings suggested that recycling of metal powder make the AM specimens softer. Subjecting the specimens made of re-used metal powder into aqueous solution, however, tends to slightly increase their hardness. The material morphology's observations complement the weight loss and mechanical testing findings. Finally, exposing the 17-4 PH AM specimens to the hypochlorite ions (OCl^-) in aqueous solution, induces corrosion which adversely affects the mechanical performance of the parts. This study has provided some bases for furthering research in the impact of using recycled AM powder of 17-4 PH steels as well as their durability in alkali solution (environment).

5. Conclusion

The current research demonstrates the effects of alkaline environment on the mechanical performances of 17-4 PH additively manufactured parts using recycled metal powder. The results indicate:

- increase in stiffness with a reduction in elastic deformation,
- reduced ultimate tensile strength and negligible change in plastic deformation,
- increased hardness,
- and finally, change in material morphology.

These changes in the mechanical performances and material morphology could be correlated to the presence of hypochlorite ions (OCl^-) in aqueous solution that can have a detrimental effect on the corrosion resistance of the 17-4 PH steels. Furthermore, this study also indicated that recycling of metal powder has strong influence on the mechanical performance of LPBF-produced parts. Such parts showed reduced tensile strength but enhanced ductility in comparison with the specimens produced using virgin 17-4 PH metal powder. In terms of future work, we propose experimentation of longer duration of exposure of LBPF specimens to the aqueous solution and study its effect on the material and mechanical behaviors.

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