The properties of stainless steel 17-4PH produced by a low-cost additive manufacturing technique, the Markforged MetalX™

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The properties of stainless steel 17-4PH produced by a low-cost additive manufacturing technique, the Markforged MetalX™

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Abstract. The lower capital and running cost of the Markforged Metal X™ platform, in comparison to competing metal additive manufacturing systems, makes it a highly attractive proposition for several industries. The unusual print, then sinter, process also has the potential to avoid many of the metallurgy issues that can arise using more typical additive manufacturing, which relies on melting to bind the material. However, the mechanical properties of the material produced must be well understood. In this paper, the properties of 17-4PH stainless steel samples produced by the Metal X™ are documented following a systematic testing program, involving standard mechanical testing as well as material characterisation techniques. Significant differences were observed between the samples tested in differing orientations. This is thought to be due to the deposition process used to lay the material prior to sintering, which results in voids and surface stress concentrations that reduce the strength of the material. Heat treatments and surface machining were both found to improve the tensile properties of the material.

1. Introduction

The Markforged MetalX™ is a proprietary metal Additive Manufacturing (AM) system, which uses a technique termed Atomic Diffusion Additive Manufacturing (ADAM). This process is best described as a hybrid between a polymer filament AM process, and the powder metallurgy of Metal Injection Moulding (MIM). The relatively low cost of the MetalX™ system may allow the uptake in industries where AM is not yet a viable process [1].

The MetalX™ system process consists of three discrete phases: print, wash, and then sinter. During the 'print' phase building material, in the form of a filament comprised of metal powder, polymer, and a binding agent, is heated and extruded onto the build plate, through a twin nozzle system to produce a 'green' part. A ceramic release filament is also applied at the interface between the part and support structure. This process is similar in method to a Fused Deposition Modelling (FDM) AM process typically used to produce polymer parts [2]. Subsequently, the artefact is 'washed' in a debinding solution to dissolve the polymer binding agent and is considered a 'brown' part in the post-washing phase.

During sintering, the components are heated in an industrial furnace under an argon atmosphere, through an initial debinding temperature at which any remaining polymeric binder is burned off, and then to a higher sintering temperature. The actual temperatures used are not revealed during processing

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and remain the intellectual property of Markforged. Following the atomic diffusion process, the metal particles diffuse together, this is claimed to form a 99.7% dense solidified artefact [3], and the ceramic supports turn from filament to powder to allow easy release of supports. Thus, leaving the cooled part ready for use or post-processing through techniques such as heat treatment or machining [4].

Although AM is often heralded as one of the cornerstones of Industry 4.0, various constraints relating to the mechanical properties of metal AM components exist, and these limitations are often incurred by the melting process of powder metallurgy techniques. The MetalX™ may avoid some of these undesirable features typical of melt-based AM processes. The sintering of fine metal powder should avoid the large columnar grains that can be seen in other AM processes. Such large irregular grains can result in anisotropic properties [5]. In addition, the layer-by-layer melting of the powder, typically results in residual stresses within the part that may adversely affect the structural integrity of the artefact in service [6]. Again, the sintering process of the MetalX™ should avoid this issue.

Although the sintering phase coalesces the powder particles together, and removes the need for a melting process, a further limitation associated with metal AM parts is still presented by the layer deposition technique used in the Metal X™ system, as this method can culminate in a stair-stepping effect of the surface topographies [7]. The heterogeneous surface finish over the built artefact gives rise to diminished mechanical properties, as stair-stepping can reduce the service life of a component subjected to dynamic loading by providing an area of high stress concentrations, whereby cracks may form and propagate [8]. As such, it is essential to evaluate the mechanical properties of the built component, prior to implementation in a critical engineering application.

This paper documents the observed properties of 17-4PH stainless steel samples generated by the Metal X™ system using standard mechanical testing methods, in conjunction with materials characterisation techniques. The differences in mechanical behaviour observed, between sample sets and defined by testing characteristics such as orientation, surface treatment, and heat treatment, is explained by discussion of the inherent macro and micro component features formed by the Metal X™ process and lays the basis of designing for AM (DfAM) using the Metal X™ system, to identify potential improvements to the manufacturing process.

2. Materials and methods

Both net shape and cylindrical specimens were used to investigate the properties of the 17-4PH stainless steel produced by the MetalX™. Computer Aided Design (CAD) software (Solidworks) was used to generate models which were then exported as a stereolithographic (STL) file, suitable for reading by the Markforged software. Tensile samples were either manufactured directly or by post AM machining. A gauge length and diameter of 27 mm and 5.05 mm respectively were used in accordance with the British Standard for tensile testing [9]. A range of orientations (with respect to the build plate) and post-processing methods were applied as shown in Table 1. For each condition, four samples were manufactured.

The initial net shape sample built vertically included supports due to the overhanging grip section (denoted by [i]). Unfortunately, these supports proved impossible to remove without damaging the gauge length. Therefore, the geometry was adapted slightly and reprinted to remove the filleted edge at either end of the gauge length, in favour of a 60° chamfer. This allowed the geometry to fit into the tensile testing rig, without incorporating supports for printing. The use of the adapted geometry for these workpieces is denoted by [*] in Table 1 and the test results. It is believed that this change will make a small difference to the results, as the gauge length section of the sample remained consistent. However, this sample was sintered in a separate furnace cycle to the other specimens.

Cylindrical specimens were machined using a CNC lathe. Following machining, attempts to further improve the surface finish of one set using an OTEC EF18 Mass Finisher were also made. Plastic conical chips were rotated at 310RPM for two hours while the samples were immersed within them. A separate sample set was heat treated at 482.2 °C (900 °F) for 1 hour followed by air-cooling.

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The surface profile of the 90° net shape machined and polished sample were recorded using a Talysurf contact measurement device. Taylor Hobson software was used to extract an average surface roughness (Ra) value for each condition.

Uniaxial tensile testing was conducted using a 5 mm/min extension rate. The small size of the specimens precluded the use of a strain gauge, but extension was recorded. Following this, the sample grips, which should have experienced limited strain, were removed in order to provide material for further analysis. Samples were mounted in Bakelite and polished to a mirror finish using standard metallographic techniques. Hardness tests using Rockwell Hardness C test conditions specified for 17-4PH stainless steel, using a diamond indenter at a load of 150N, were performed. As the hardness test is performed on the internal geometry of the part, the influence of machining and polishing can be neglected.

Fractography of the broken tensile specimens was conducted using an FEI Inspect S50. This Scanning Electron Microscope (SEM) was also used to examine the microporosity in the metallurgically prepared samples.

Table 1. Sample Manufacturing Plan.

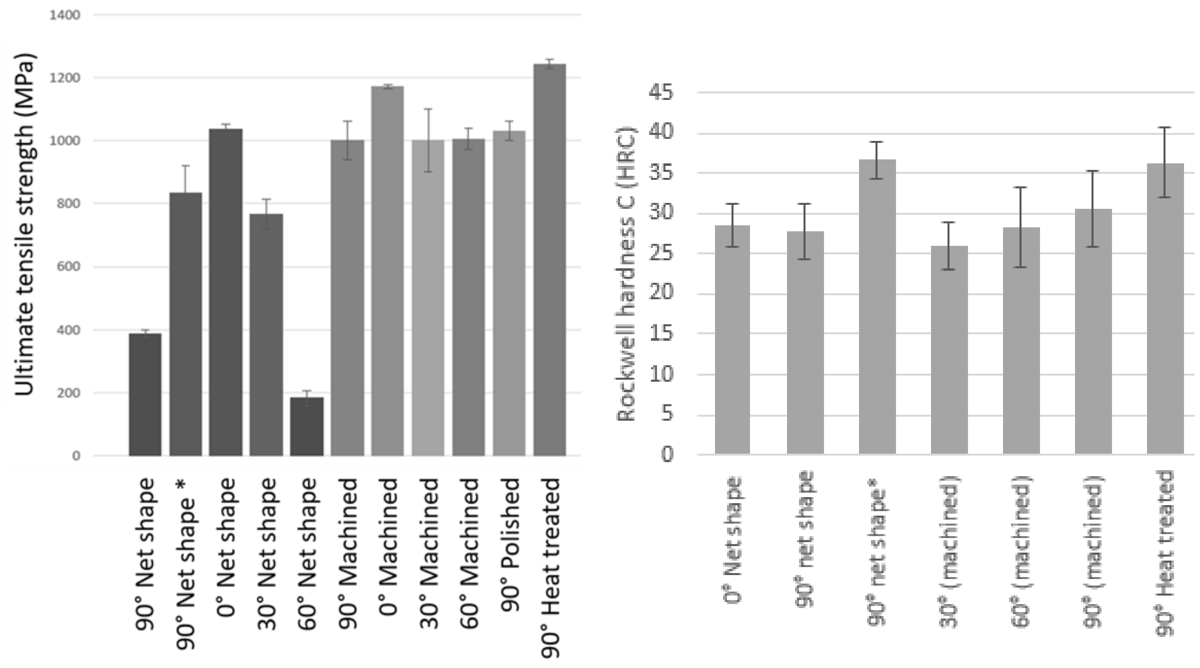
Initial Geometry	Orientation	Machined	Polished	Heat Treated
Net shape	0° (horizontal)	N	N	N
Net shape	30°	N	N	N
Net shape	60°	N	N	N
Net shape[i]	90° (vertical)	N	N	N
Cylinder	0° (horizontal)	Y	N	N
Cylinder	30°	Y	N	N
Cylinder	60°	Y	N	N
Cylinder	90° (vertical)	Y	N	N
Cylinder	90° (vertical)	Y	Y	N
Cylinder	90° (vertical)	Y	N	Y
Net shape* (modified)	90° (vertical)	N	N	N

3. Results

Tensile testing revealed little to no plastic deformation, with samples apparently de-forming elastically until failure. The mean and standard deviation in Ultimate Tensile Strength (UTS) for each condition is shown in Figure 1a. The two records for 90° net shape are due to the need for geometry modification as discussed in the method section. The sample marked with the * indicates the modified geometry.

When testing specimens manufactured net shape, significant differences were observed between the orientations tested. In contrast, machined samples tested at 30°, 60° and 90° showed a very similar UTS. In both cases the samples tested at 0° to the build plate had the highest strength. Polishing the surface resulted in a slight increase to the UTS, but a much larger improvement was noted when the sample was heat treated.

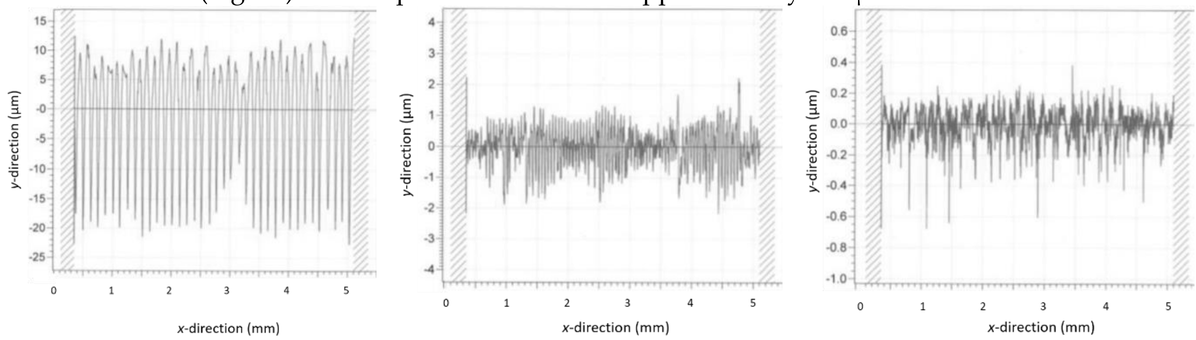
The variation in Rockwell hardness is shown in Figure 1b. Similar results were found in all cases except the modified net shape (indicated with the *) and heat-treated specimen, both in the vertical (90°) orientation.



(**a**) (**b**)

Figure 1. Mean mechanical properties recorded for each sample set. Error bars indicates the standard deviation. (a) UTS, and (b) Rockwell hardness.

The change in surface profile after machining and polishing tensile specimens can be observed in Figure 2. It is clear that each step reduced the variation in surface profile, and corresponding reductions in Ra were recorded at 7.40 μm, 0.49 μm and 0.08 μm. The repeating peaks and troughs visible in the as-built condition (Fig 2.a) had a repetition distance of approximately 100 μm.



(**a**) (**b**) (**c**)

Figure 2. Surface profile of samples in the (a) as-built, (b) machined and (c) polished condition.

SEM examination of the metallurgically prepared bulk material revealed small pores apparently randomly positioned within the material (Figure 3a). No difference was observed between material in the 0° and 90° orientation. The SEM images were used to calculate a relative density of 96.7 ± 0.3 % and 96.4 ± 0.2 % in the 0° and 90° orientations, respectively.

However, SEM analysis of the fracture surface revealed large, very high aspect ratio, voids dominating the fracture surface appearance (Figure 3b, c, d). These were arranged in lines and orientated perpendicular to the build direction.

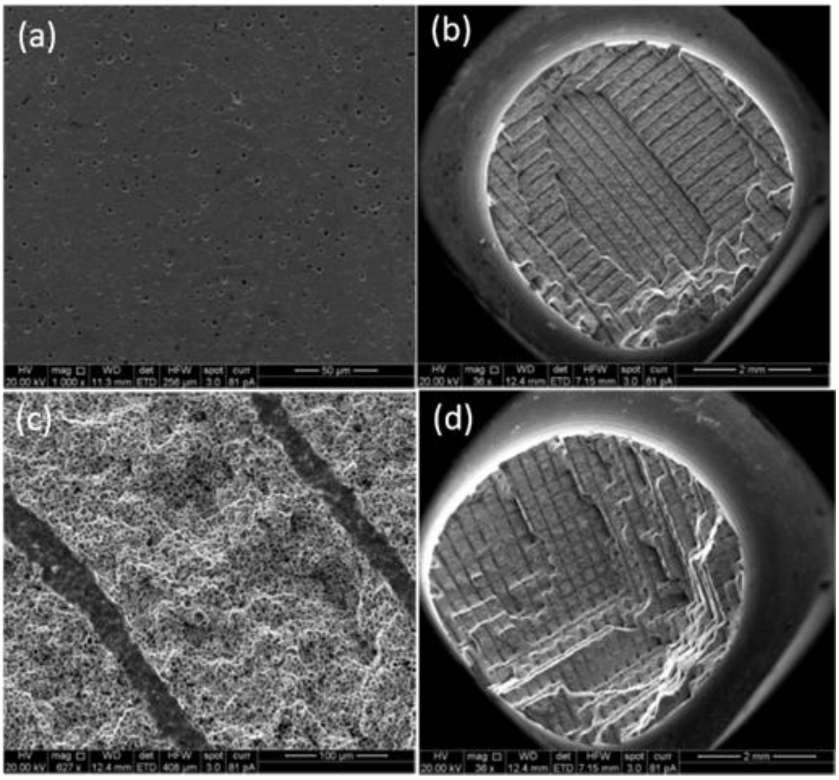


Figure 3. SEM observations of (a) bulk material from grips of the 0° tensile sample, (b) 90° machined fracture surface, (c) higher resolution image of 90° fracture surface and (d) 60° fracture surface.

4. Discussion

The variation in UTS between orientations is disappointing given that the sintering process should have resulted in a homogenous microstructure. However, some of the variations in UTS observed can be explained based on the other results recorded. In particular, given that the difference between most orientation's UTS is significantly reduced by post build machining, we can infer that the surface roughness plays a significant role in reducing the UTS. The UTS was increased as the surface roughness fell for the vertical specimens tested as-built, machined, and polished.

The "staircase" geometry introduced by the FDM process is known to reduce the strength of the components [7,8]. This can help to explain the fact that samples tested at 30° or 60° had a lower strength than those at either 0° or 90°, where there is unlikely to be such an effect.

Most of the as-built roughness is likely to result from the material deposition during the FDM process. The periodic variation in the profile (Fig. 3.a) matches the expected thickness of the layers after sintering (100 µm). Therefore, efforts could be made to improve the properties by modifying the material deposition parameters to minimise roughness.

It is likely the deposition of material has also affected the internal structure of the component. The large voids on the fracture surface have an appearance that suggests they were formed by insufficient overlap between extrusion tracks during the FDM material deposition. It can be observed that the voids change orientation on the fracture surface, which corresponds to the different orientations used during the FDM process. It is clear that, under loading, these voids not only reduced the area available to take the load, but also resulted in significant stress concentrations. The fracture surface suggests that samples fractured at the interface between layers where these voids will align. These large voids will have also contributed to the limited ductility observed in all samples. This is further evidence that the properties of the MetalX™ material could be enhanced by the modification of the FDM process.

Another concerning observation is the significant difference in hardness between the initial 90° net shape specimen and the modified geometry 90° specimen. The small change in the geometry is unlikely to have had any detectable impact on the hardness of the material. However, the second specimen was subjected to a different sinter cycle to the rest of the specimens. Given that the sinter temperature is not known, and indeed the entire cycle is set by Markforged, it may be that there is inconsistency between cycles. This has implications for the qualification of the MetalX™ material for industries that demand traceable manufacturing processes.

5. Conclusions

The tensile properties of stainless steel 17-4PH samples, manufactured by the low-cost AM system (MetalX™), have been evaluated. Significant differences were observed between samples tested in different orientations. This has been attributed to be primarily because of the surface roughness introduced during the initial FDM deposition phase. Large voids, thought to be introduced during the FDM phase, will have further reduced the material properties. A difference in hardness was observed between similar samples subjected to nominally the same sinter cycle, which needs further testing to establish the source of this inconsistency. The “black box” nature of the process means we are, at present, unable to determine if there were any differences between the thermal cycles experienced by the two samples.

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