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Enhancement of an additive-manufactured austenitic stainless steel by post-manufacture heat-treatment

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Abstract:

The effect of post-manufacture heat-treatment on the mechanical strength of an additively-manufactured austenitic stainless steel has been investigated. Microstructural investigations revealed that the as-manufactured material exhibited a multi-scale structure, composed of grains, cells, dislocations and nano-sized particles. Annealing at 400°C resulted in a 10% increase in yield strength, associated with the additional precipitation of a population of nano-sized silicates. Annealing at higher temperatures resulted in a decrease in strength, attributed primarily to the thermal instability of the cell structure in the as-manufactured material. The results demonstrate that by careful control of annealing conditions the structure and mechanical properties of additively-manufactured austenitic stainless steel can be optimized by post-manufacture heat-treatment.

Keywords: Additive manufacturing; 316L stainless steel; heat-treatment; mechanical properties; Orowan strengthening

1. Introduction

Additive manufacturing (AM) is a rapidly developing technology, combining computer-aided design with material processing and forming to allow the creation of solid objects [1-10]. Among the various AM methods, selective laser melting (SLM) is of increasing interest for the manufacture of engineering materials. In this method a high-energy laser beam is used to melt locally a bed of metal powder according to a pre-determined design, resulting in the manufacture of a two-dimensional cross-section [8-10], with solid parts achieved by repeating this process layer-by-layer from bottom to top to form the final part [8, 11]. This “bottom-up” AM method allows the production of complex structural parts that cannot be achieved by traditional processing methods.

316L stainless steel (316L SS) is an austenitic composition with high strength and good corrosion resistance, widely used in the aerospace, energy, and automotive industries, as well as in medical devices [3, 8]. Due both to its relatively high machining costs, and to its high sensitivity to grain boundary corrosion after welding, there is increasing interest in the manufacture of 316L SS parts by AM, through which near-net-shape components can be produced.

In addition to optimization of the process parameters during AM, the mechanical properties and performance of additively-manufactured components can also be modified by post-manufacture heat-treatment. For example, the occurrence of phase transformations has been reported in additively-manufactured 316L SS after annealing at high temperature [4]. A key point, however, is that the high strength and good ductility of additively-manufactured 316L SS has been attributed in part to the presence of a dislocation network structure [12]. As annealing at elevated temperatures can result in a reduction in dislocation density via thermally-activated recovery processes, this may therefore result in a deterioration in mechanical properties. Accordingly, it is necessary to study carefully the link between the thermal stability of the dislocation

network structure and changes in mechanical properties during annealing of additively-manufactured 316L SS.

2. Experimental

The material used in this study is an additively-manufactured 316L SS, produced by the SLM method. Detailed information regarding the material is given in [12]. Samples for investigation were cut by spark machining, mechanically polished, and then annealed for 1 h at various temperatures up to 800°C, followed in each case by water cooling.

Micro-hardness was measured using a load of 1 kg and a dwell time of 10 s. Ten measurements were performed on each sample. Dog-bone shaped tensile specimens with a gauge size of 8 mm in length, 2 mm in width and 1 mm in thickness, were cut with the tensile axis perpendicular to the build direction. The samples were tested at a constant strain rate of $4 \times 10^{-4} \text{ s}^{-1}$.

Microstructural characterization was carried out using an Oxford AZtec electron backscattered diffraction (EBSD) detector attached to a JEOL 7800F scanning electron microscope (SEM), and a JEOL 2100 transmission electron microscope (TEM). Samples for EBSD were mechanically polished followed by electropolishing at 20 V for 10 s in an electrolyte of perchloric acid and acetic acid (1:4 ratio) at room temperature (RT). Analysis of the EBSD data was carried out using the Oxford Instruments Channel 5 software package. Specimens for TEM investigation were ground to approximately 50 μm thickness, and then thinned by jet-polishing using a voltage of 50 V at -20°C in an 5% perchloric acid/ethanol solution. The chemical composition was analyzed using energy-dispersive X-ray spectroscopy (EDS) in a FEI TECNAI G2 F20 TEM.

3. Results and discussion

Fig. 1(a) shows the micro-hardness of the additively manufactured 316L SS as a function of heat-treatment temperature. The micro-hardness of the as-manufactured material is 252 HV. With increasing annealing temperature, the micro-hardness first

gradually increases to a maximum value of 291 HV at 400°C, and then continuously decreases to 228 HV at 800°C. Fig. 1(b) shows tensile curves of the as-manufactured sample and of samples annealed at 400°C and 800°C for 1 h. The yield strength and uniform elongation in the as-manufactured condition are 586 MPa and 35%, respectively. After annealing at 400°C for 1 h, the yield strength increases to 642 MPa, while the uniform elongation only slightly decreases to 32%. After annealing at 800°C for 1 h, the yield strength decreases to 482 MPa and the uniform elongation is restored to 35%.

Fig. 1 (a) Micro-hardness of additively-manufactured 316L SS after annealing at various temperatures for 1 h; (b) engineering stress-strain curves of the as-manufactured 316L SS sample and of samples annealed at 400°C and 800°C for 1 h.

Fig. 2 shows example EBSD orientation maps of the as-manufactured sample and of samples annealed at 400°C and 800°C for 1 h. The EBSD maps reveal a microstructure typical of additively-manufactured material, namely a checkerboard macrostructure in the section perpendicular to the build direction (as shown in Figs. 2(a-c)), and a columnar grain structure in sections containing the build direction (as seen in Figs. 2(d-f)) [12]. This microstructural appearance has been attributed to rapid melting and solidification away from equilibrium conditions during the AM process [4, 12]. Grain sizes were determined from the EBSD maps using an area reconstruction method with a 2° grain boundary definition, and are given in Table 1 as equal circular

diameter values. The average grain size in the as-manufactured sample is about 5.9 μm , with only a small variation observed after annealing at either 400°C or 800°C.

Fig. 2 EBSD orientation maps of the as-manufactured sample (a, b), and of samples annealed at 400°C (c, d) and 800°C (e, f) for 1 h. The microstructure in the section taken perpendicular to the build direction is shown in (a, c, e), while (b, d, f) show a section containing the build direction. White lines represent misorientations $\geq 2^\circ$ and $< 15^\circ$, with black lines showing misorientations $\geq 15^\circ$.

Fig. 3 shows TEM images of the as-manufactured sample and of samples annealed at 400°C and 800°C for 1 h. As shown in Fig. 3(a) and 3(b), a characteristic cell structure is seen in the as-manufactured sample, similar to that seen previously in additively-manufactured 316L SS [6, 12]. In some regions cellular sub-grains are observed (Fig. 3(a)), whereas other regions are composed of columnar sub-grains (Fig. 3(b)) [7]. The average cell diameter, as determined from TEM images showing cells with an equiaxed morphology, is about 0.48 μm (see Table 1), with a columnar length of more than 10 μm . Many dislocations are also observed within the cells.

Table 1 Microstructural parameters and calculated Orowan strengthening contributions

Sample	as-manufactured	400 °C/1 h	800 °C/1 h
Average grain size, μm	5.9	5.6	5.4
Average cell diameter, μm	0.48	0.50	0.46
Volume fraction of particles (f), %	0.42	1.38	0.61
Average particle size (d), nm	64	21	14
Average particle spacing (λ), μm	10.1	0.97	1.49
Orowan strengthening ($\Delta\sigma$), MPa	6	61	40

After annealing at 400°C for 1 h, both the cell structure and the high density of cell-interior dislocations are preserved (Figs. 3(c) and 2(d)), with almost no detectable change in the average cell diameter (see Table 1). The characteristic cell structure of the as-manufactured sample is therefore thermally stable at temperatures up to 400°C. However, after annealing at 800°C for 1 h (Figs. 2(e) and 2(f)), a significant change in the dislocation structure is observed, with the microstructural appearance changing to be more typical of that seen in 316L SS after plastic deformation at elevated temperatures [13]. Interestingly, however, the length scale of the dislocation cell structure remains almost unchanged.

For each sample five areas were characterized using the TEM observations to determine both the volume fraction and average size of silicates. The results are listed in Table 1. It is seen that in the as-manufactured sample the volume fraction of silicates is only 0.42%, and that these silicates have a relatively large average size. After annealing at 400°C for 1 h, the volume fraction of silicates increases to 1.36%, indicating that precipitation of silicates takes place during annealing at 400°C. The newly precipitated silicates are small in size, resulting in a decrease in the average silicate particle size. After annealing at 800°C, some dissolution of the silicates takes place, resulting in a decrease in the volume fraction to 0.61%, accompanied by a small decrease in the average particle size.

Fig. 3 TEM images of the as-manufactured sample (a, b), and of samples annealed at 400°C (c, d) and 800°C (e, f) for 1 h. Examples of cell structures are shown in micrographs (a), (c) and (e), while examples of columnar structures are shown in (b), (d) and (f).

The contribution to the tensile yield strength of the silicates ($\Delta\sigma$) can be estimated according to the Orowan mechanism, as described by:

$$\Delta\sigma = M \cdot \Delta\tau = M \cdot \frac{G \cdot b}{\lambda} \quad (1)$$

where M is the Taylor factor, τ the shear stress, G the shear modulus, b the Burgers vector, and λ the average spacing of the silicate particles. Here, λ is determined using the equation:

$$\lambda = \frac{4(1-f)r}{3f} \quad (2)$$

where f is the volume fraction, and r is the average radius of the silicate particles. Based on these equations, and using the values given in Table 1, the strength contributions due to the silicate particles have been estimated, taking values of $M = 3.06$, $G = 77.2$ GPa and $b = 0.256$ nm. The results are given in Table 1. It is seen that the silicate particles

contribute very little to the yield strength of the as-manufactured sample, due both to their small volume fraction and the large average particle size. However, in the sample annealed at 400°C for 1 h the newly precipitated silicates contribute about 55 MPa to the yield strength – almost completely accounting for the increase in strength of the as-manufactured sample after annealing at this temperature (56 MPa).

Fig. 4 Example EDS mapping showing the presence of silicates in the sample annealed at 400°C for 1 h. (a) TEM image; (b), (c), (d), (e) and (f) are element distribution maps of Fe, Cr, Si, O and Mn, respectively.

Interestingly, based on the microstructural parameters the silicate particles are predicted to give a strength contribution of 34 MPa after annealing at 800°C for 1 h. Annealing at 800°C for 1 h in fact results, however, in a decrease of the yield strength of 104 MPa compared to the as-manufactured sample. Moreover, the length scale of the dislocation cell structure in this condition is comparable to that of both the as-manufactured sample and of the sample annealed at 400°C for 1 h (as seen in Table 1). The appearance of the cell structure is, however, very different in the sample annealed

at 800°C, indicating that at this temperature significant modifications to the dislocation arrangements forming the cell structure have taken place.

These observations highlight the importance of preserving the dislocation cell structure of the as-manufactured material during any subsequent heat-treatment, and shows additionally that a window of annealing temperatures exists where the cell structure can be preserved while also taking advantage of other strengthening mechanisms. The exact origin of the strength contribution from the dislocation cell structure is not yet fully understood, and further studies to examine the specific changes in the dislocation arrangements in the cell structure that take place during annealing are necessary.

A consideration that should be taken into account in this regard is that annealing of 316L SS may introduce sensitization of the grain boundaries. A preliminary study has shown, for example, that rapid attack on the boundaries of melting pools in additively manufactured 316L SS may occur after annealing at 650°C for 1h [15]. Therefore, it is also important to study the consequence of annealing on the corrosion behavior of additively manufactured 316L SS, even for low-carbon compositions that are traditionally considered as resistant to sensitization. Preliminary oxalic acid tests according the ASTM A262 Practice A [16] on the 316L SS material of this study show no sensitization of grain boundaries of the present as-manufactured and of the 800°C annealed samples [see supplementary material].

The results of the present study show that additive manufacturing of 316L SS results in a multi-scale structure. On the melt pool scale, a checkerboard pattern is formed, consisting of grains with size of up to tens of micrometers, separated in many cases by high angle boundaries. Inside these grains, a cell structure is developed, where the average cell size is a few hundred nanometers. Within these cells, a high density of dislocations and a population of nano-sized silicate particles are found. This multi-scale structure results in a yield strength of the as-additively manufactured 316L SS of 586 MPa, very close to that reported elsewhere [6, 12]. During annealing at temperatures up to 400°C fine silicate particles are precipitated, while the cell structure and dislocations are stable. This results in a gradual increase in micro-hardness and yield

strength. However, annealing at higher temperatures leads to a continuous decrease in micro-hardness and yield strength, despite the continued presence of a population of silicate particles, suggesting that at temperatures higher than 400°C a transformation of cell/dislocation structure take place, resulting in a reduced strength contribution. This conclusion is supported by the observation of a change in the appearance of the cell structure in the sample annealed at 800°C.

4. Conclusions

The microstructural stability and mechanical properties of additively manufactured 316L SS during post-manufacture heat treatment have been studied by annealing at temperatures up to 800°C for 1 h. Annealing at temperatures up to 400°C leads to an 10% increase in yield strength compared to the as-manufactured state, from 586 MPa to 642 MPa, with only a small loss in uniform elongation. This is attributed to the thermal stability of the cell/dislocation structure, in combination with precipitation of nano-sized silicate particles. The preservation of the cell/dislocation structure allows the high strength and good ductility of the as-manufactured material to be maintained, while the silicates contribute via an Orowan mechanism to the majority of the observed strength increase of 56 MPa in the sample annealed at 400 °C for 1 h. Annealing at temperatures higher than 400°C results, however, in a continuous decrease in the strength of the material, as the cell structure is no longer stable, and some dissolution of the silicates takes place.

The present study shows that by careful control of the annealing conditions the mechanical properties of additively-manufactured 316L SS can be further increased by post-manufacture heat treatment, providing a route for enhancement in mechanical properties of additively-manufactured 316L SS parts in addition to optimization of the AM processing parameters. The results also highlight the importance of understanding the strength contribution and thermal stability of the cell structure formed during additive manufacturing, and how these relate to the nature of the dislocation

arrangements in the as-manufactured and annealed conditions. Although the results presented here are for 316L SS, the findings of the present study can be also applied to other heat-treatable metallic materials produced using additive manufacturing, thus pointing to a new avenue for tailoring the mechanical properties of additive-manufactured parts by controlled heat-treatments.

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