Strengthening of 304L Stainless Steel by Addition of Yttrium Oxide and Grain Refinement during Selective Laser Melting

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Abstract

This study investigates the role of submicron yttrium oxide on the microstructural evolution and mechanical properties of selective laser melted 304L oxide dispersion strengthening (ODS) nanocomposite. 304L SS powder and a mixture of 304L SS powder and yttria nanoparticles were used as two feedstocks for fabricating parts using selective laser melting (SLM) process. Scanning electron microscopy and energy-dispersive X-ray spectroscopy revealed a homogenous distribution of Si-Mn-O nanoparticles in the SLM 304L matrix and Y-Si-O nanoparticles in the SLM 304L ODS alloy. Electron backscatter diffraction images imply that the addition of yttria disrupts the formation of large columnar grains in SLM 304L, resulting in the formation of finer equiaxed grains. The average microhardness values increased from 240 HV in SLM 304L to 305 HV in SLM 304L ODS alloy, due to the combined effects of grain refinement and dispersion hardening.

Keywords: selective laser melting, nanocomposite, oxide dispersion strengthened alloy, 304L stainless steel

Introduction

Austenitic stainless steel (SS) alloys have numerous industrial applications due to high corrosion and oxidation resistance at room temperatures. However, limited yield strength at elevated temperatures (>600°C) limit their advanced engineering applications to lower temperatures [1,2]. Strengthening of austenitic stainless steels can promote their extensive adoption in high-temperature applications. Several efforts have been devoted to enhance mechanical properties of various materials such as aluminum and stainless steels by introducing second phase such as particulate ceramic [3-5]. In recent years, metal matrix nanocomposites have been introduced as thermally stable dispersed nanoparticles in the metal matrix [6]. Similarly, an important approach for strengthening steel alloys is oxide dispersion strengthening (ODS), which involves the dispersion of yttrium oxide nanoparticles in the metal matrix [7]. Many size-dependent properties of nanoparticles are quite different from the properties of bulk materials due to large surface area and confinement effects, which can consequently lead to enhancement of mechanical properties [8]. The very high number density of uniformly dispersed Y- or Y–Ti oxide nano precipitates in a steel matrix can reduce the creep rate at 650-900°C and increase the tensile

strength of materials [9]. However, the major utilized techniques for fabrication of strengthened alloys have been casting and mechanical alloying, which is a nonequilibrium solid-state powder metallurgy process [6]. Despite the relatively expensive and time-consuming conventional approaches, additive manufacturing (AM) is a promising technology to manufacture high-resolution components at a lower cost and higher speed [10].

AM is a technology for fabricating complex geometrical parts directly from computer-aideddesign (CAD). In every technique of AM, given the 3D CAD model is sliced into 2D profiles. Each 2D layer is then added to the previous layer until the 3D object is complete [11]. AM technology offers the fabrication of parts from a wide range of materials, including metals, ceramics, and polymers [12]. Fabrication of metallic parts is possible via powder bed, powderfeed, and wire-feed AM techniques [13]. Selective laser melting (SLM), which is a powder-based AM technology enables manufacturing of complex lattice structure of metallic parts that were difficult to manufacture using conventional techniques [14]. SLM involves laser melting of metal powder bed followed by rapid solidification [15]. Higher resolution and better dimensional accuracy are remarkable advantages of SLM over other metal fabrication techniques of AM [16]. A problem of fabricating strengthened metal alloys involving nanoparticles during casting is the tendency of nanoparticles to agglomerate that leads to inhomogeneous microstructure. Rapid solidification of SLM favorably results in microstructural refinement, which can potentially hinder solute segregation and assist particle dispersion [6]. Thus, the fabrication of ODS alloys using SLM is significantly beneficial in terms of microstructural refinement.

Nguyen et al. [17] fabricated near full density SS 304L parts using SLM. Fine grains (about 4 μ m) consisting of nano-cellular and martensite phase were observed. Guan et al. [18] investigated the impact of layer thickness on the mechanical properties of SS 304L manufactured by SLM. Results revealed that the best mechanical properties of 694 MPa in ultimate tensile strength (UTS), 541 MPa in 0.2% yield strength (YS), and 39% in elongation were achieved at the optimal layer thickness of 40 μ m.

Kim et al. [19] fabricated austenitic 316L ODS steel using powder metallurgy processes. The UTS of 660 MPa was obtained and the microstructural analysis only confirmed the presence of $Y_2Si_2O_7$ and TiO₂ particles with a size of a few hundreds of nanometers. Zhou et al. [20] reported enhanced UTS of 940 MPa in HIPed austenitic ODS steel containing Y-Ti-Si oxide dispersoids with the size of 17 nm. Suresh et al. [21] developed 316L ODS austenitic steel by addition of 0.35 wt% Y₂O₃ using mechanical alloying and hot extrusion consolidation followed by solutionizing at 1150 °C for 1 h. Traverse and longitudinal sections of the sample contained fine equiaxed grains and elongated grains with a mean size of 280 ± 5 and 440 ± 9 nm, respectively. A high density of dispersed Y-Ti-O nanoparticles with a mean size of 9 nm, and a low fraction of coarser nanoparticles of Y-Si-O were observed. Enhanced hardness of 346±25 HV in this alloy was attributed to the presence of dispersed oxide nanoparticles and grain refinement. Kotan et al. [22] developed nanocrystalline SS 316L and SS 316L+1.5 wt.% yttrium by mechanical milling at cryogenic temperature. Structure of both alloys consisted of γ -austenite and α' -martensite phases with a grain size of about 19 nm. After annealing, SS 316L revealed grain growth up to micron

sizes, while SS 316L with 1.5 wt% yttrium demonstrated stabilized microstructure with the grain size of ~116 nm.

Boegelein et al. [23] fabricated thin-walled builds using SLM process with as-mechanically alloyed ODS-PM2000; reporting the as-grown builds had inferior YS (YS=500 MPa) compared to conventional PM2000 alloy (YS=800 MPa). In another work by Ryan et al. [24] on additively manufactured MA956 ODS using selective laser sintering process, the UTS of 651 MPa (65% of the wrought strength) for the density of 97% was reported.

This work aims to investigate the effect of oxide nanoparticle dispersion on the microstructural evolutions and mechanical properties of SLM-processed 304L SS ODS alloy. For this purpose, typical 304L SS powder and 304L SS ODS alloy (304L+5 wt% Yttria) were manufactured using the SLM process, and characterized for their microstructure and mechanical properties. The objective of this work is to evaluate SLM process as a promising direction towards AM of ODS alloys.

Experimental Procedure

The powder feedstock in this study was Sandvik Osprey gas-atomized AISI 304L austenitic SS powder (<45 μ m) with the chemical composition given in Table 1, and H.C. Starck yttria (<1 μ m). In order to prepare the precursor of 304L ODS alloy, the 304L powder was mixed with 5 wt.% of Yttria in a planetary ball mill for 4 hours using the ball to powder ratio of 5:1. Then, the mixed powder was loaded to the SLM machine for fabricating the 304L ODS material. The 5 wt.% yttria ratio was selected in order to be easily detected using different characterization techniques.

Table 1. Chemical composition of 304L stainless steel powder (wt.%)

Element	Cr	Ni	Fe	С	Si	Mn	Р	S	Ν
wt.%	18.853	10.060	Bal	0.017	0.720	1.3	0.012	0.005	0.083

To print the samples using SLM machine, an ORLAS Creator Metal 3D printer (OR Laser/ Coherent) with 250 W Yb: YAG fiber laser and a wavelength of 1067 nm was used. A set of twelve cylinders with a dimension of R4×8 mm were additively manufactured using the 304L powder and mixed 304L+5 wt.% yttria, with following parameters; laser power 105 W, scan speed 50-600 mm/s with an increment of 50, spot size 50 μ m, hatch spacing 50 μ m, layer thickness 30 μ m and scan rotation angle of 105°. The oxygen level in the SLM chamber was consistently kept below 100 ppm by continuously purging pure nitrogen.

After the SLM process, Archimedes method was used to measure the relative density of samples. Phase identification was conducted by using X-ray diffraction (XRD) analysis (Bruker AXS D8 Discover), operating with Cu K_{α} radiation at 40 kV and 20 ranged from 20-100°. Sample preparation for microstructural characterizations was accomplished according to standard metallographic procedures. Polished samples were electroetched for 15 s at an applied DC voltage of 15V using an electrolyte solution of 10 wt.% oxalic acid. Then, microstructural characterization

of samples was performed using optical microscopy (Zeiss, Axiotron) together with scanning electron microscopy (SEM, FEI Quanta 3D) on the cross-sections parallel to build direction for both SLM 304L and SLM 304L+5 wt.% yttria.

Electron backscattered diffraction (EBSD) was conducted on vibratory polished samples with 50 nm diamond suspension for 8 hours. The EBSD data collection was performed using an Orientation Imaging Microscopy (OIM) Data Collection 7.2 software in FEI Quanta 3D operating at 15 kV accelerating voltage, 1.7 nA beam current and a step size of 50 nm. The collected data were then processed using OIM Analyses 7.2 software. Microhardness of the SLM 304L and SLM 304L+5 wt.% Yttria samples was measured using a microhardness tester (Leco, LM-248AT) at a load of 500g. The reported values in the results were the average values from 10 random indentations for each type of samples.

Results and Discussion

The morphology of 304L, yttria powder particles, and the mixture of 304L with 5 wt.% yttria after light ball milling are shown in Figure 1(a) and Figure 1(b), respectively. As shown in Figure 1(a), gas atomized 304L powder comprises spherical particles with some satellites attached to larger particles and the mean particle size of 33.3 μ m. The yttria particles had a prismatic shape with the mean particle size of 0.968 μ m, as shown in the inset of Figure 1(a). Most of the particles contained their original spherical shape while few particles found to be deformed during the ball milling. The inset in Figure 1(b) displays a closer look at the surface of lightly ball milled powder at a higher magnification.



Figure. 1. Morphology of (a) 304L and yttria nanoparticles (the inset) and (b) the mixed 304+5 wt.% yttria powder

Figure 2 displays the X-ray diffraction analysis that was conducted to evaluate the crystal structure of the mixed powder of 304L+5 wt.%, SLM 304L specimen and SLM 304L+5 wt.% yttria. The main peak of mixed 304L+5 wt.% yttria was austenite phase with a small trace of ferrite. Moreover, due to the high amount of added yttria (5 wt.%), the yttria peak was also detected in XRD pattern; indicating that the yttria did not get dissolved in the matrix due to the light ball milling process. In contrast to the mixed powder, XRD analyses on SLM 304L and SLM 304L+5

wt.% yttria, revealed that the ferrite phase has emerged as the peak with the highest intensity; suggesting a strong texture of this phase after SLM process. The high cooling rate during the SLM process caused an incomplete transformation of ferrite to austenite phase [25]. No peak from yttria was detected after the SLM process, suggesting dissolution of yttria nanoparticles within the ferritic matrix.



Figure 2. XRD pattern of mixed 304L+5 wt.% yttria powder (purple), SLM 304L (red) and SLM 304L+5 wt.% yttria (green)

The relative densities of SLM 304L and SLM 304L+5 wt.% yttria with different scan speeds are depicted in Figure 3. Overall, relative density decreased by increasing the scan speed. It is mainly attributed to the appearance of lack-of-fusion voids associated with the lower volumetric energy density of laser at higher scan speeds. However, the SLM 304L demonstrated higher relative density compared to the SLM 304+5 wt.% yttria. The highest relative density of 99% and 96.5% were achieved for SLM 304L and SLM 304+5 wt.% yttria, respectively. Lower relative density of SLM 304L+5 wt.% yttria could be related to the interruption of uniform powder layering by yttria particles. The non-uniform layering then resulted in more lack-of-fusion pores and consequently lower relative density in the SLM manufactured parts.



Figure 3. The relation between relative density of SLM 304L and SLM 304L+5 wt.% yttria with laser scan speed

The optical micrographs of cross-section parallel to the build direction of SLM 304L and SLM 304L+5 wt.% yttria samples after electro-etching is presented in Figure 4. In Figure 4 (a), the typical melt pool boundaries of the SLM process is evident. Moreover, the bright contrast in Figure 4 (a) is the epitaxial columnar grains which grew in the opposite direction of heat flow. During SLM process, the appearance of epitaxial grain growth is favorable due to the effect of same crystallographic orientation as well as identical chemistry of melt and underneath solidified layer which leads to heterogeneous nucleation with no barriers [26]. As shown in Figure 4 (a), no evidence of porosities, balling effect or cracks between layers were observed in the micrograph of SLM 304L+5 wt.% yttria shown in Figure 4 (b) revealed the formation of large porosities and balling effect between layers. It is noteworthy that, the epitaxial columnar grains (bright contrast) could not be detected in the cross-section parallel to the build direction; suggesting the formation of more equiaxed grains in SLM 304L+5 wt.% compared to SLM 304L with columnar grains.



Figure 4. Optical micrograph from cross-section parallel to the building direction (a) SLM 304L and (b) SLM 304L+5 wt.% yttria

The SEM micrograph of SLM 304L and SLM 304L+5 wt.% yttria are shown in Figure 5. The typical cellular substructure, in both SLM 304 and SLM 304L+5 wt.% yttria is formed due to rapid solidification of the SLM process. Generally, at a high cooling rate of 10^3 - 10^7 C/s cellular substructure forms due to lack of sufficient time for the growth of the secondary dendrite arms [26]. Figure 5 (a) demonstrates the precipitation of small spherical particles within the matrix in the SLM 304 sample ranged from 20 to 80 nm. In a similar study, Ghayoor et al. [25] reported these nanoparticles to be a compound of Si-Mn-O that were formed due to the reaction of available Si and Mn in the matrix (Si=0.72 wt.% and Mn=1.3 wt.%) with the available oxygen in the chamber.

Figure 5 (b) shows the SEM micrograph of SLM 304L+5 wt.% yttria at a higher magnification. The SLM 304L+5 wt.% yttria comprised several spherical nanoparticles within the matrix. The diameter of these fully spherical nanoparticles was in the range of 10-150 nm. The nanoparticles were uniformly dispersed throughout the entire sample with a little regional variation in the density of the nanoparticles. It has been reported by Ghayoor et al. [27] that these

nanoparticles were a compound of Y-Si-O; being more stable at high temperature compared to yttria nanoparticles.



Figure 5. SEM micrograph of (a) SLM 304L and (b) SLM 304L+5 wt.% yttria, V=200 mm/s and after electroetching

Figure 6 illustrates the microstructure of SLM 304L and SLM 304L+5 wt.% yttria crosssectioned perpendicular to the build direction in the EBSD grain map. As shown in Figure 6 (a), the microstructure of SLM 304L contained columnar grains with the average grain size of $7\pm 6 \mu m$. However, microstructure of SLM 304L+5 wt.% yttria consisted of more equiaxed grains with a finer average grain size of $3.7\pm 3 \mu m$, compared to SLM 304L; indicating the role of yttria nanoparticles as potential inoculants in facilitating the heterogeneous nucleation and formation of finer equiaxed grains. This grain refinement in SLM 304L+5 wt.% yttria could enhance the mechanical properties of the material due to the Hall-Petch strengthening mechanism [28].



Figure 6. EBSD grain map micrograph of (a) SLM 304L and (b) SLM 304L+5 wt.% yttria, V=200 mm/s, perpendicular to building direction

Figure 7 shows the microhardness values for SLM 304L and SLM 304L+5 wt.% yttria at different scan speeds; the microhardness values dropped by increasing the scan speed, possibly due to lower densities associated with the higher scan speeds. The average microhardness values for SLM 304L and SLM 304L+5 wt.% yttria were 240 ± 7 HV and 305 ± 12 HV, respectively, which were relatively higher than the microhardness of annealed 304L (210 HV) [29]. Therefore, the addition of yttria could enhance the mechanical properties of SLM 304L+5 wt.% yttria compared to SLM 304L. This increase in hardness can be related to the combined effect of the homogenous distribution of nanoparticles through dispersion mechanism (Orowan) [30] as well as grain refinement through the Hall-Petch mechanism [28].



Figure 7. Microhardness values of SLM 304L, SLM 304L ODS alloy, and 304L in the annealed condition [29]

Summary and Conclusion

In this study, the role of adding yttria nanoparticles in strengthening of 304L matrix was investigated. In this sense, the light ball milling was adopted to mix 304L with yttria powder as the precursor for additively manufacturing of 304L ODS alloy, and the following conclusion can be drawn:

- 1) The SLM 304L+5 wt.% yttria showed lower density (max=96.5%) compared to SLM 304L (max=99%) due to the relatively higher lack of fusion and non-uniform layering.
- 2) OM and EBSD micrograph had shown the yttria particles altered the growth of columnar grains in SLM 304L with the grain size of 7±6 μ m toward more finer equiaxed grains in SLM 304L+5 wt.% yttria with the grain size of 3.7±3 μ m.
- 3) SEM micrograph revealed the formation of cellular substructure in addition to the formation of fine nanoparticles in both SLM 304L and SLM 304L+5 wt.% yttria
- 4) Adding yttria to 304L matrix substantially increased the microhardness value from 240±7 HV for SLM 304L to 305±12 HV for SLM 304L+5 wt.% yttria due to the combined effect of dispersion and grain refinement mechanisms.

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