Microstructure and mechanical property of 12CrNi2 high strength steel fabricated by laser additive manufacturing technology

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Abstract. 12CrNi2 high strength steel thin wall with few defects was fabricated by laser additive manufacturing technology. The microstructure of as-built sample was studied using optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Additionally, the mechanical property was also investigated by microhardness tests. It was found that the microstructure of the as-built sample mainly consists of columnar dendrites aligned in building direction because of the fast directional cooling during LAM. Cellular grains, and equiaxial dendrites are also observed in different regions of the as-built sample due to heat accumulation effect. Furthermore, many types of microstructure, such as tempered martensite, M-A constituent, retained austenite, lower and upper bainitic ferrite, are appeared in the as-built sample resulting from complex heat history and wide temperature range during LAM process. The maximum and minimum microhardness (499 HV0.2 and 272 HV0.2) occur in the top and bottom region respectively, while microhardness of the middle region with value of 452 HV0.2 just slightly reduces compared to the top section of as-built sample.

1. Introduction
Laser additive manufacturing technology (LAM), an advanced additive manufacturing process using laser beam as energy resource, is the most frequently used processing method due to the excellent advantages such as high energy density, high temperature gradient, and less processing time [1]. According to the different laser power and forming methods, LAM can be divided into different types: laser solid forming (LSM), selective laser melting (SLM), selective laser sintering (SLS), and laser engineered net shaping (LENS) [2-5]. Various metals or alloys can be fabricated from powder or wire by LAM in a layer-by-layer pattern, and these as-built components always have high density and excellent mechanical properties [6]. Thus, there is an increasingly strong demand for industry to use LAM and develop data base to produce parts, especially those with complex shape.
Common, researchers have reported that many types of alloys, such as 316L steel, M2 steels, H13/Cu components, Ti-6Al-4V, titanium alloys, Inconel 625, Inconel 718, Ni-based superalloys and Co-based alloys, have been fabricated by LAM in the fields of aerospace, chemistry and nuclear. Unfortunately, the variety of commercially available materials for application in LAM is still limited so far [6, 7].

High strength steel has excellent strength-toughness performance and is widely used as structural materials, such as pressure vessels, gears, brake discs and those used in shipbuilding industry [8]. However, most of researchers focus on the various wielding technology of high strength steel and there are few publications about the additive manufacturing technology of high strength steel [9-11]. In the present work, LAM technology was used to fabricate 12CrNi2 high strength steel thin wall. Many material characterization methods were carried to investigate the microstructure and mechanical property of the as-built part.

2. Experimental procedures

The material used in the present study was 12CrNi2 high strength steel powders with size of ~250µm and spherical shape. These powders were fabricated by School of Materials Science and Engineering, Northeastern University (China). The chemical compositions of the powders were listed in Table 1.

| Elements | C    | Ni   | Cr   | Mn | Si | V   | Mo | S   | P   | O   | Fe  |
|----------|------|------|------|----|----|-----|----|-----|-----|-----|-----|
| Content  | 0.064| 2.01 | 1.53 | 1.0| 0.06| 0.46| 0.70| 0.008| 0.010| 0.043| Bal |

The LAM of 12CrNi2 high strength steel thin wall was performed with a LAM system consisting of a 10 kW fiber laser, a powder feeding unit and an argon atmosphere chamber which can prevent the as-built parts from being oxidized effectively. The 12CrNi2 high strength steel thin wall was fabricated on a Q235 plate with dimensions of 150mm×120mm×12mm (length×width×height).

Figure 1 shows the schematic diagram of LAM 12CrNi2 high strength steel process. It is obvious that the component was produced as layer-by-layer pattern. During the forming procedure, the alloy powder was delivered to a molten pool created by laser beam with high purity argon gas, and then rapidly solidified onto the former layer. The as-built thin wall with dimensions of 100mm×12mm×18mm (length×width×height) was formed with a constant laser power, 2.5kW, and the laser scanned speed was kept 6 mm/s. One thing should be mentioned is that all the powders must be dried over 2h in a drying furnace before LAM process.
Small sample was machined by wire electric discharging cutting for investigation of microstructure and mechanical properties and then experienced standard mechanical grinding and polishing processes. The sample was acquired perpendicular to the laser scanned direction with a whole height of 12mm. Next, the metallographic sample was etched in a mixed solution of 1mL nitric acid + 19mL absolute alcohol. The microstructure of the as-built sample was observed by an OLYMPUS optical microscopy (OM) and a Philips Quant 200 scanning electron microscopy (SEM) equipped with an energy disperse X-ray spectroscopy (EDS) which was used to analyze the chemical compositions of some micro-regions. The micro-hardness of the as-built sample was also investigated using a HXZ-1000 Vickers micro-hardness tester under the load of 200g with a dwell time of 15s.

3. Results and discussion

3.1. Microstructure of the as-built sample

Figure 2 shows the microstructure of the as-built sample fabricated by LAM. It is obvious that different regions of the as-built sample present different microstructural characteristics. In the bottom region, a number of columnar dendrites with around 9.7µm width are observed, as can be seen in figure 2(a). Scan track boundaries appear between building layers, and many small cellular grains are shown in the transition interface region existing between scan track boundaries. Besides, a small number of keyhole pores can be also observed in the as-built sample, which may occur in case of a high energy input and thus a very deep melt depth, resulting in the solidification of the melt on the previous layers before the gap can be fully filled [12].

Figure 2. Optical micrograph of the microstructure of the as-built sample: (a) the bottom region; (b) the middle region; (c) the top region.

Note: SDA-second dendrites arms, IFZ-incomplete fusion zones, STB-scan track boundary.

With respect to the middle region, many long columnar dendrites grow epitaxially along the building direction, which is similar to the bottom region, as shown in figure 2(b). This phenomenon is
mainly attributed to the typical characteristic of heat flow in the LAM process because during LAM process, components are fabricated layer-by-layer. The heat is predominately dissipated from the molten pool to the pre-layers or the substrate. As a result, the directional columnar dendrites always grow from the bottom to the top, almost parallel to the building direction. The width of these grains is about 16.6µm, larger than that of the columnar dendrites observed in the bottom region. This increase occurs due to the high temperature gradient and rapid cooling rate during the initial built layers. However, the temperature gradient and cooling rate will gradually decrease with the built layers increasing, resulting from the heat cycle history and heat accumulation effect. Another thing should be mentioned is that some incomplete fusion zones are observed in the figure 2(b), indicating the process parameters should be further optimized.

The equiaxial dendrites appear predominately in the top region, and a few cellular grains exist beside the dendrites, as presented in the figure 2(c), and this type of dendrites can also be seen around the edge of the middle region (figure 2b). This similarity was observed because the cooling rate and temperature decrease significantly and grains have enough time to grow, resulting in the appearance of equiaxial dendrites. Besides, the inter-dendrites spacing has increased to 40µm, much larger than that in the bottom and middle regions, which can also demonstrate the decline of temperature gradient and cooling rate.

Figure 3. SEM micrograph of the microstructure of the as-built sample: (a) the bottom region; (b) the middle region; (c) the top region.

Note: M'-tempered martensite, M-A-martensite-austenite constituent, RA-retained austenite, LB-lower bainite, GB-grain boundary, UBF-upper bainitic ferrite, PF-proeutectoid ferrite.
Figure 3 presents the microstructural characteristics of the as-built sample from bottom to the top region. It can be observed that a great number of tempered martensites (M') exist in the initial layers, as can be seen in figure 3(a). The length of lath M’ ranges from 5µm to 10µm. Some Martensite-Austenite (M-A) constituents with average size of 5.4µm are visible close to the primary grain boundary and retained austenite (RA) is also found in this region. Besides, a few needle-like lower bainitic (LB) ferrites with the length of approximate 8.6µm and width of about 50nm are also observed in the columnar dendrites, which grow from primary GB towards the grain interior. As for the middle region, a plenty of LB ferrites with length ranging from 8µm to 16µm are presence next to a few lath-like upper bainitic ferrites (UBF) which have a longer average length of 15µm, as presented in figure 3(b). Moreover, it can be seen that many fine granular bainites fill up the inter-LB region. When it comes to the top region, lath-like UBF and coarse irregular proeutectoid ferrite (PF) are predominate, as shown in figure 3(c). It is obvious that the forming of as-built microstructure suffers a very complex heat history and experiences a very wide temperature range, and this conclusion is similar as the results which have been reported in Ref. [13].

3.2. Mechanical property of the as-built sample
Microhardness distribution of the as-built sample about the distance from sample surface is shown in figure 4. Overall, typical Vickers microindentation hardness of the as-built sample is increasing with the growing distance from sample surface. Furthermore, the average microhardness of as-built sample is in the order of top region > middle region > bottom region with the value of 499 HV$_{0.2}$, 452 HV$_{0.2}$, and 272 HV$_{0.2}$ respectively. The maximum microhardness occurring in top region is attributed to appearance of abundant lath-like UBF, which shows excellent mechanical properties. Microhardness of the as-built sample experiences a slight decrease in the middle region, resulting from the fewer UBF and fine granular bainites. However, the overwhelming majority of M' in the bottom region lead to the minimum microhardness of the as-built sample.

![Figure 4. Microhardness distribution of the as-built sample about the distance from sample surface.](image)

4. Conclusions
The 12CrNi2 high strength steel thin wall with few defects was successfully fabricated by LAM. Conclusions can be summarized as follows.

(1) The microstructure of as-built sample mainly consists of columnar dendrites, cellular grains, and equiaxial dendrites. And different regions show different microstructural characteristics.

(2) Complex heat history and wide temperature range in the LAM process lead to many types of microstructure in the as-built sample. M', RA, LB, UBF, granular bainite, and PF can be observed in the bottom, middle, and top region respectively.
(3) Microhardness of the as-built sample is increasing with the growing distance from sample surface, and the average value is measured as 499 HV₀.₂, 452 HV₀.₂, and 272 HV₀.₂ at the top, middle, and bottom section respectively.

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