Characterization of Microstructure in Additively Manufactured Stainless Steels

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1. Introduction

3D printing, which has received worldwide attention, refers to a process of manufacturing a three-dimensional solid object through stacking materials. 3D printing, also known as Additive Manufacturing (AM) in the official ASTM terms, is possible to implement complex design by stacking materials in contrast to the conventional subtractive machining such as Computer Numerical Control (CNC) machining.

Metal AM is a technique to build a 3D shape by stacking metallic materials using energy sources such as laser or electron beam. It has been attracting attention in the fields of medical, aerospace, nuclear industry and many more. In order to replace existing parts with additively manufactured parts, their reliability must be verified.

In this study, we investigated the microstructural characteristics of 304L stainless steel, which is widely used for nuclear reactor structures. This material is fabricated by PBF (Powder Bed Fusion) and DED (Directed Energy Deposition) which are two representative fabrication methods of metal AM.

2. Additive Manufacturing Methods

AM is a process of joining materials (metal, ceramic, and polymer) to make objects from 3D model data, usually layer upon layer, as opposed to subtractive machining that is conventional methodology [1]. There are two popular processes for metal AM, which are PBF and DED. The various AM processes have their own characteristics. In this section, we examine the features of each process.

2.1 Powdered Bed Fusion (PBF) Methods

PBF is an AM process in which thermal energy selectively fuses regions of a powder bed [1]. The powder bed fusion selectively dissolves the section of the 3D model by applying a thin powder to the bed and scanning a heat source such as a laser or electron beam [2]. When one section is completed, the chamber is lowered down by the thickness of the powder layer then thinly coated and scanned again. This process is repeated until the entire 3D form is completed. The approximate structure and melting process of the PBF machine is shown in Fig.1.



Fig. 1. Powder bed fusion process and machine scheme [3].

PBF has high level of complexity and good surface finish and minimum post-processing due to powder acts as support material [1]. The disadvantages of PBF are relatively low stacking density and slow speed.

2.2 Direct Energy Deposition (DED) Methods

DED is a kind of AM processes in which focused thermal energy is used to fuse materials by melting as they are being deposited [1]. DED is a process where metal power or wire is combined with an energy source to deposit material onto a build tray or an existing part directly. Because DED is very similar to the welding process, it is specifically used to create 3D structure from metals and alloys [4]. The process of DED as the nozzle moving along the path is shown in Fig.2.



Fig. 2. Schematic of the Direct Energy Deposition (DED) process [5].

The nozzle of DED machine can move in multiple

directions since it is attached on a multi axis arm. Therefore, DED is free to move and does not require a support structure. It makes DED applicable to large parts and effective in repairing and adding parts. The disadvantages of the DED are that the surface condition of manufactured parts is coarse and the accuracy is low, which is required for post-processing [4].

3. Experimental Procedure

3.1 3D Sample Preparation

Two kinds of metal powders were prepared. The material used in the PBF was 304L powder supplied by CSC(창성). The 304L powder has a particle size of 15 to 45 μ m and an apparent density of 3.97 g/cm³. The chemical composition of the powder provided by CSC is given in Table 1.

Table.1. Chemical composition of 304L powder used in PBF process, wt. %

	Cr	Ni	Mn	Si	С
STS304L	19.4	10.3	0.98	0.54	0.028

The material used in the DED was micro-melt 304 supplied by CARPENTER. The micro-melt 304 powder has a particle size of 45 to 150 μ m and an apparent density of 4.34 g/cm³. The chemical composition of the powder provided by CARPENTER is listed in Table 2.

Table.2. Chemical composition of micro-melt 304 powder used in PBF, wt. %

	Cr	Ni	Mn	Si	С
Micro- Melt304	18.4	9.7	1.4	0.67	0.025

The 3D printing samples were fabricated under optimized deposition conditions for bulk characterization at 'KAMI' for PBF and 'InssTek' for DED.

3.2 Specimen Preparation

To investigate the surface of the sample, specimens were taken from the side as shown in Fig.3. Then we observed the surface with an Optical Microscope (OM) and a Scanning Electron Microscope (SEM).

To check the pores on the surface, the specimens were ground and polished, and the surface pores were observed by SEM.

To observe the microstructure of the longitudinal cross-section, the specimens were vibratory polished and observed by SEM. The 304L specimens manufactured by PBF and DED were etched by Aqua regia reagent (HNO₃: HCl=1:3) and observed with OM.



Fig. 3. Schematic of sample taken from bulk

4. Results

4.1 Surface Image

The appearances of the samples were observed using an OM and SEM. The pattern can be seen with the naked eye which are shown in Fig.4a for PBF and Fig.4b for DED. The SEM image of the PBF (Fig.4e) shows an oblique pattern. While, the pattern on the surface is not visible at the SEM image of the DED (Fig.4f). This is caused by the size of the laser spot. Since PBF has laser spot size of several tens of μ m, it can be seen in a low magnification SEM image. On the other hand, DED has about 1 mm in laser spot size, so it is not exposed in the SEM image.



Fig. 4. Top surface images of specimens manufactured by (a) PBF and (b) DED, scanning patterns of (c) PBF and (d) DED, SEM images showing top surface of samples manufactured by (e) PBF and (f) DED.

4.2 Internal Pore

The pores on the internal surface of the PBF specimen are randomly distributed with irregular shapes of tens of μ m in size. We could find some big-size pores shown in Fig. 5(a), which are believed to be process induced defects. The pores on the surface of the DED specimen (Fig.5b) are distributed about 50 per 1,000 μ m² with circular shapes of hundreds of nm in size. This circular pores are believed to be induced by gas from the powder.



Fig. 5. SEM images of pores on the surface of (a) PBF and (b) DED.

4.3 Microstructure

The microstructure of longitudinal cross-section was observed by SEM and OM. The SEM and OM image of the PBF sample are shown in Fig.6a & Fig.6c, respectively. We observed elongated grains extending in the building direction of the manufacturing process. Fig. 6b & Fig.6d are the SEM and OM image of the DED sample, respectively. The semi-circular patterns are shown in Fig.6d, which are caused by local re-melting of the layers formed in the previous steps. There are also found in other AM samples [6].



Fig. 6. SEM images of (a) PBF and (b) DED specimen with vibratory polishing, OM images of sample produced by (c) PBF (etched with aqua regia reagent) and (d) DED (etched with aqua regia reagent)

5. Summary

In this work the microstructural characteristics of 304L specimens made by PBF and DED processes were investigated. Tentatively, the following results could be obtained.

- 1. The size of melting spots in the DED is larger than that in the PBF.
- 2. Pores, which are expected to be caused by process defects or charged gases, were found on the internal surface.
- 3. Semi-circular patterns were observed in the DED sample, which are signs of local re-melting.

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