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SELECTIVE LASER MELTING OF PURE TIN: MICROSTRUCTURE STUDY

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Abstract

Selective Laser Melting (SLM) of pure tin was examined and 3D samples were successfully fabricated. High relative density of 99.9 % was achieved. Microstructure study was carried out via a combination of X-ray diffraction (XRD), light microscopy (LOM) and electron backscatter diffraction (EBSD). Under LOM, columnar structures, some spanning the entire height of the sample, are observed in the vertical plane. This study also examines the variation of relative density against process parameters such as hatch spacing and the input volumetric energy density for the SLM of tin.

KEYWORDS: Selective laser melting, Tin

Introduction

Selective Laser Melting (SLM) is an Additive Manufacturing (AM) technology that is gaining popularity due to its ability to fabricate near full density metallic components without the need for post processing (Chua and Leong 2014). In recent years, industrial interest in direct manufacturing with AM is evident in the increase in global revenue for AM products and services. AM metals increased by 50 % in 2014 to 48.7 million USD and made up 42.6 % of the total products and services revenues from AM, previously dominated by polymer-based materials (Wohlers 2015). AM of metals is a sector predicted to continue a high pace of growth as the technology matures.

The SLM process is carried out in an enclosed chamber with a platform movable in the z-axis. It starts with coating a thin layer of powder on a substrate plate. A high power laser is then deployed to melt selective areas according to computer aided design (CAD) data which has been pre-processed into scanning vectors information for individual layers. Once the first layer has been processed, the platform lowers by a pre-determined distance (20 – 100 µm) and a new layer of powder is coated on to the preceding layer (Yap, Chua et al. 2015). The laser is deployed again to process the second layer. This combination of platform lowering, powder coating and laser scanning is repeated until the desire components are completely fabricated.

Tin is a metal known for its low melting point of 505 K, with a density of 7.298 g/cm^3 in the β-phase. Hence, it is widely used as a solder in the form of Sn-Pb alloy. Tin is also corrosion resistant and is often used in plating to protect iron based alloys that would rust easily otherwise.

As a metal, tin is used to make decorative housewares such as punched tin lanterns. Recent studies have also shown that pure tin, when coupled with a conductive polymer, can provide high specific capacity and high cycling stability for Na+ cells (Komaba, Matsuura et al. 2012, Dai, Zhao et al. 2014). Although tin has low melting point and is easily processed compared to other metals and alloys, there has been little research on the AM of tin. In this study, SLM processing of tin was investigated and process parameters were optimized to fabricate near full density samples.

Materials and Methods

Powder Material

The tin powder used in this study is purchased from TLS Technik GmbH. The powder particles are spherical in shape, as shown in Figure 1, with a D90 of 45 µm. Spherical powders were chosen to for their improved powder flowability over their irregular-shaped counterparts. Energy-dispersive X-ray spectroscopy (EDXS) analysis has shown that the powder material has a purity of 99.8 %.

SLM 250 HL

The SLM machine used in this study is the SLM®250HL. It is equipped with a Yb: YAG fibre laser with a wavelength of 1.06 µm, a maximum power of 400 W and a Gaussian energy distribution profile. The substrate area is 250 mm × 250 mm and the platform can move 300 mm in the z-direction. The building chamber is flushed with argon gas until the oxygen level in the chamber is lower than 0.2 % before the fabrication process starts. This is to minimize the oxidation of metals at elevated temperatures.

Experimental Procedure

The experiments were carried out at a fixed scanning speed of 3,000 mm/s and a fixed layer thickness of 50 µm. Laser power was varied from 100 W to 200 W and hatch spacing ranged from 0.1 mm to 0.2 mm. Samples of 10 mm × 10 mm × 10 mm were built. Near-full density samples were obtained and details on the parameter-density relationship have been discussed in an earlier study (Yap, Chua et al. 2016). This paper discusses the analysis of SLM processed tin samples via XRD, light microscopy and EBSD.

Microstructure and Discussion

X-ray Diffraction (XRD)

XRD was carried out with the Empyrean, PANalytical. Analysis showed some changes in the relative composition of crystal orientations between the SLM processed tin and the tin powder (Figure 1). The 4 main peaks of the powder material, at 2θ of 30.66 °, 32.04 °, 44.94 ° and 62.58 °, correspond to the crystallographic planes of {2 0 0}, {1 0 1}, {2 1 1} and {1 1 2} respectively. Whereas the 4 main peaks of the SLM processed tin are at 2θ of 44.94 °, 62.58 °, 79.59 ° and 95.26 °, corresponding to the crystallographic planes of {2 1 1}, {1 1 2}, {3 1 2} and {1 0 3} respectively. XRD analysis of the tin powder also suggests a significant but relatively small amount of amorphous structure in the powder material.

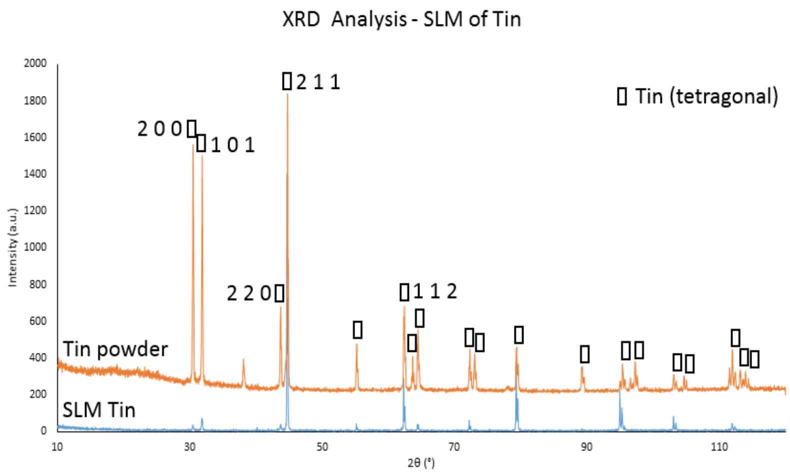

Figure 1. X-ray Diffraction analysis of tin powder and SLM processed tin powder. The analysis shows different relative peaks for the two materials.

XRD analysis suggests that the SLM process favours the formation of particular crystal orientations while significantly reducing others. For instance, the amount of {2 0 0} and {1 0 1} planes were drastically reduced when compared to the analysis of the powder material. Moreover, there was no amorphous structure in the SLM processed samples.

Light optical microscopy (LOM)

The samples were polished and subsequently etched for 120 s in a solution of hydrochloric acid and methanol in the ratio 1:49. Pores can be seen in Figure 2 (left). The XY plane and the XZ plane did not exhibit any characteristic microstructures seen in SLM processed parts such as those in AlSi10Mg or M2 tool steel. The grains seemed to have a random distribution in the XY plane. In the XZ plane, there were also little traces of the melt pool formed during the SLM process, which was evident in alloys. Instead, the cross section observation revealed elongated columnar structures. A fraction of these columnar structures stretched from the bottom to the top of the sample.

Furthermore, there were also zig-zag patterns on the grains near the sides of the samples (Figure 2, right). Twinned grains and recrystallized grains could also be observed. These deformations were the results of working during the polishing process and tin is extremely susceptible to such phenomenon as it has a low hardness and a low crystallization temperature of 231.9 °C. Diagonal grains at the sides of powder bed based fusion parts has been reported in literature (Antonysamy, Meyer et al. 2013). However, in their study, the diagonal grains are the results of powder nucleation and have an upward slope from outside, which are in line with the temperature gradient during the fabrication process. In SLM processed tin, the diagonal grains were observed to have a downward slope from the outside instead.

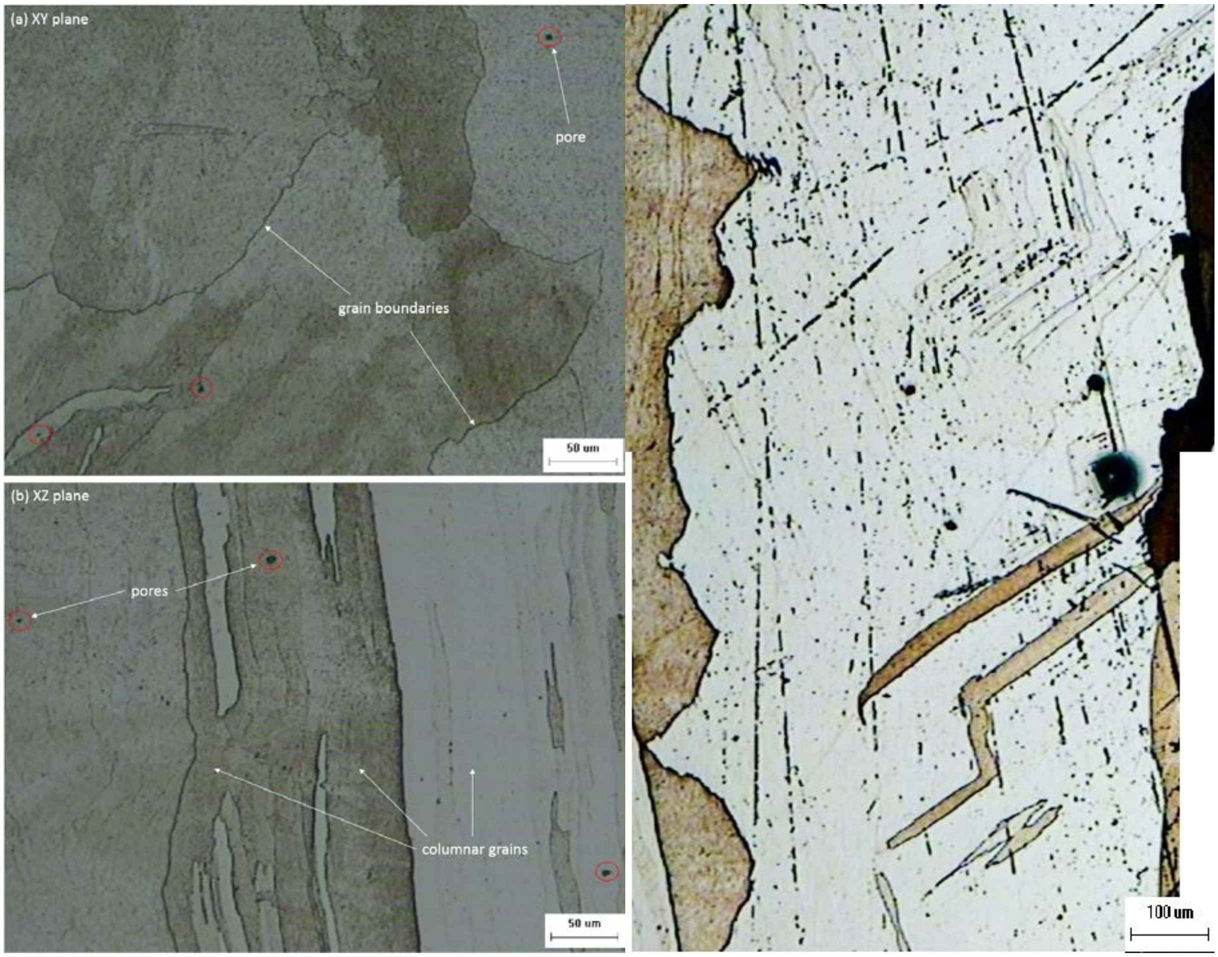

Figure 2. Optical microscopy images of SLM processed tin at a lower magnification, showing XY planes (a) and XZ planes (b), with pores circled in red. On the right, composite cross section (XZ plane) image of SLM processed tin near the edge, showing the zig-zag structure of the columnar crystal, twinned grains and recrystallized grains within original grain boundaries that result from working during polishing.

Electron backscatter diffraction (EBSD)

EBSD analysis was also carried out alongside with SEM to examine the local crystal orientation in the samples. In Figure 3, the high angle grain boundaries (HAGBs, misorientation > 15 °) are shown in black and low angle grain boundaries (LAGBs, misorientation < 15 °) in grey. The EBSD analysis confirmed the columnar structures observed via LOM. Moreover, EBSD analyses have also shown that SLM processed tin has preferred grain orientation, which matches indications from the XRD analysis shown earlier. In both the XY plane and the XZ plane, most of the crystals were in the <1 1 0> orientation, as shown by a larger proportion of blue coloured grains in both maps.

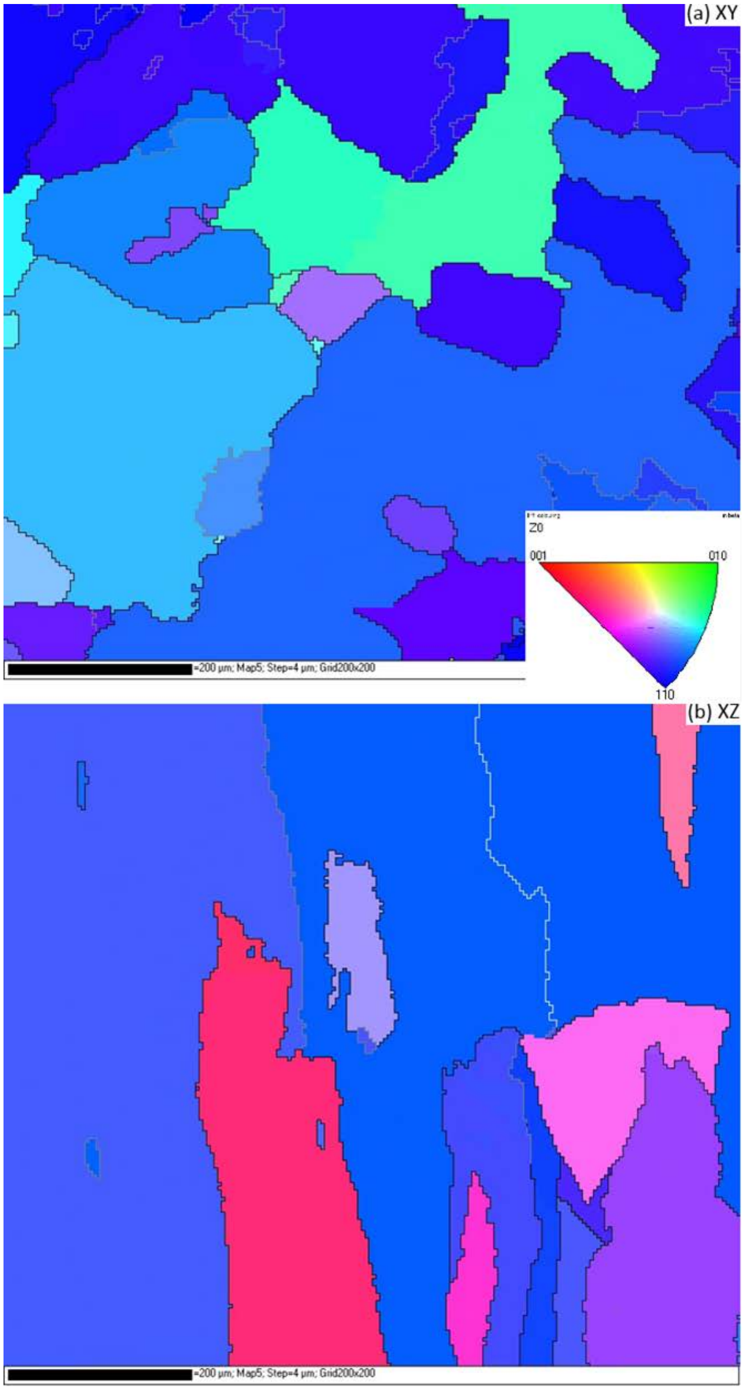

Figure 3. EBSD analysis of SLM processed tin, displaying the different crystal orientations: <0 0 1> in red, <0 1 0> in green and <1 1 0> in blue.

The viability of tin for use in the SLM process was verified in an earlier study. The SLM process produces tin with columnar structures and the crystal grains are anisotropic, with preferred grain orientations. Further research can be done to determine the cause of preferential grain growth and mechanical properties of SLM processed tin.

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