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Metallurgy of high-silicon steel parts produced using Selective Laser Melting



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ABSTRACT

The metallurgy of high-silicon steel (6.9wt.Si) processed using Selective Laser Melting (SLM) is presented for the first time in this study. High-silicon steel has great potential as a soft magnetic alloy, but its employment has been limited due to its poor workability. The effect of SLM-processing on the metallurgy of the alloy is investigated in this work using microscopy, X-Ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD). XRD analysis suggests that the SLM high-silicon steel is a single ferritic phase (solid solution), with no sign of phase ordering. This is expected to have beneficial effects on the material properties, since ordering has been shown to make silicon steels more brittle and electrically conductive. For near-fully dense samples, columnar grains with a high aspect ratio and oriented along the build direction are found. Most importantly, a $\langle 001 \rangle$ fibre-texture along the build direction can be changed into a cube-texture when the qualitative shape of the melt-pool is altered (from shallow to deep) by increasing the energy input of the scanning laser. This feature could potentially open the path to the manufacture of three-dimensional grain-oriented high-silicon steels for electromechanical applications. © 2016 Acta Materialia Inc. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

1. Introduction

Over the past few years, Additive Manufacturing (AM) technologies have been enjoying an ever-growing success, thanks to the unrivalled design freedom they offer for the fabrication of both plastic and metallic components [1]. In particular, Selective Laser Melting (SLM) is emerging across a broad range of sectors, including automotive, medical and aerospace, for the creation of functional metallic parts. SLM achieves densities comparable to the bulk material by applying a high-power laser beam to a layer of metallic powder, according to the data contained inside the CAD file of the part. The laser energy fully melts the powders together, consequently creating solid metal upon cooling.

The use of laser-based AM has been investigated for processing a range of metallic materials for structural applications, including stainless and carbon steels, as well as a number of titanium, cobalt and aluminium alloys [2–5]. However, the potential of this manufacturing technology for applications other than structural is almost unexplored. A significant example in this regard is represented by those materials with ferromagnetic properties, such as

soft and permanent magnets. Applications that rely upon such materials include the large family of devices that transform electrical energy into mechanical energy, such as motors, and from mechanical to electrical, such as AC generators. The contribution of Zhang et al. [6] is the sole example of the application of SLM for processing a soft magnetic alloy. In their paper, the authors showed the beneficial effect of microstructure refinement by SLM-processing on the DC soft magnetic properties of Permalloy. The lack of further reference on the topic indicates a gap in the literature, as it seems that the successful processing of magnetic core materials through AM has the potential to open the path to a new generation of designs for highly performing electromechanical devices.

An important class of soft magnetic materials are silicon steels, which are iron alloys with silicon in concentrations ranging from 2–7wt. Within the family of silicon steels, Fe-6.5Si alloys have a high potential in magnetic device applications due to their high electrical resistivity, near-zero magnetostriction, and low magnetocrystalline anisotropy [7]. Despite their high potential, these alloys are rarely used in commercial applications because of their very limited ductility, which results in poor workability. A recent technique developed by JFE Steel employs a chemical vapour deposition treatment to add silicon once the lamination has been created [8]. The two 6.5Si lamination types obtained through this

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technique, namely JNEX and JNHF, represent the only examples of commercialised high-silicon electrical steels. Other attempts of high-silicon steel processing have been explored in the literature, in the form of powder cores [9], cast strips [10] and electron-beam physical vapour deposited sheets [11].

The tool-free nature of SLM could offer an alternative manufacturing route for processing near-fully dense high-silicon steel parts with unique microstructural properties. Importantly, owing to the additive nature of the SLM process and the thermal history the material experiences, a crystallographic texture can be induced. A number of contributions have shown that SLM processing can create interesting crystallographic features [12,13]. In Refs. [12], a high power laser was employed to produce highly anisotropic stainless steel with strong morphological and crystallographic textures. In Refs. [13], it was shown that changing the scan strategy (i.e., the path followed by the processing laser) affects the crystallographic texture of AlSi10 Mg. The authors showed that a strong fibre texture obtained by scanning every layer using unidirectional vectors could be changed into a weak cube texture when scan vectors rotated by 90° within and between layers were employed. As regards to the silicon steels, controlling the crystallographic texture is very attractive in a number of applications, since the <001> crystallographic direction is the “easy axis” of magnetisation [14]. While cold rolling is a widespread method to obtain laminations with strong <001> texture along the rolling direction and <101> texture along the transverse and normal directions (cube-on-edge or Goss texture, as explained in Ref. [15]), a manufacturing process able to produce cube-textured parts could potentially extend the applicability range of the alloy.

The aim of this paper is to show that three-dimensional high-silicon steel parts can be manufactured using SLM and to present their metallurgy. First, the contribution of laser energy density is studied with the goal of promoting part densification. Then, the microstructure is investigated in terms of solid phase formation, grain size and morphology, as well as crystallographic texture.

2. Experimental methods

2.1. Sample manufacturing and post-processing

High-silicon steel powder with approximately 6.9% wt. Si (refer to Table 1 for a list of the alloying elements) was provided by LPW Technology Ltd., Runcorn, UK. It was experimentally verified that the powder particles are approximately spherical in shape with a symmetrical size distribution centred at 35 µm. Particle size ranges from approximately 10 µm–60 µm. This is in fairly good agreement with the range declared by the supplier (15 µm–45 µm). Powder was first dried at 60 °C for 120 min and then processed to produce test samples using a Realizer SLM-50 (Realizer GmbH, Borcheln, Germany), equipped with a continuous 100 W yttrium fibre laser (YLM-100-AC). The SLM process was conducted under an Argon atmosphere with an oxygen level below 0.4%. The platform on which the specimens were built was kept at 200 °C to maintain the part at an elevated temperature so as to reduce thermal expansion gradients. Standard cubic samples of dimensions 5 × 5 × 5 mm were produced for analysing the porosity, crack formation and the

microstructures.

As regards the manufacturing process, the main SLM parameters are defined in Fig. 1. In the present study, laser power (P), laser beam focus position, hatch-spacing, scan distance and layer thickness were kept constant. The values chosen for these parameters are those recommended by the Realizer SLM-50 manufacturer for processing stainless steel powders. Among the alloys routinely processed using the Realizer SLM-50 (which include Co-Cr, AlSi10 Mg and Ti6Al4V), the thermal and optical properties of atomised stainless steel powder are expected to be the closest to those of silicon steel, owing to the similar iron content in the two alloys. The scan speed (v) was varied in order to study the effects of the energy input $E = P/v$ on the material properties. As regards the scan strategy, each layer was scanned once using vectors oriented along either the X-axis (scan direction-x, SD_x) or the Y-axis (scan direction-y, SD_y), alternatively. This strategy is referred to as bidirectional X-Y in the present manuscript. Table 2 summarises the process parameter values used in this study. For simplicity, each sample is identified by the values of energy input E used to build it. For instance, the sample manufactured using $P = 70$ W and $v = 0.5$ m/s, which corresponds to an energy input $E = 140$ J/m is referred to as S_{140} .

2.2. Study of sample metallurgy

In order to investigate the porosity and microstructural features, the manufactured 5 × 5 × 5 mm cubic samples were cut along three perpendicular planes, parallel to the cube faces. The test specimens were cross-sectioned, mounted, and polished. The relative porosity was determined from the optical micrographs of the vertical cross-sections of the samples. An Eclipse LV100ND microscope (Nikon, Tokyo, Japan) was used to obtain the micrographs. As a quantitative means of assessing crack formation, the cumulative crack length (c.c.l.) was used. This parameter was defined as $c.c.l. = 1/n \sum l_{ij}$, where l_{ij} is the length of the j th crack on the i th polished sample surface and n is the total number of sample surfaces considered for each sample. To achieve repeatability, for each test sample a minimum of four to a maximum of six cross-sectional optical micrographs were acquired and analysed using the open source image processing software ImageJ [16].

A TM3030 scanning electron microscope (SEM) by Hitachi, Tokyo, Japan, with a 20 kV accelerating voltage and backscattered electron detector was used to evaluate the microstructures. In order to assess grain morphology and size, SEM micrographs were acquired from both the horizontal (top) and vertical (front and side) cross-sections of each sample. In order to visualise melt-pool boundaries, the sample surfaces were etched by exposing them to a solution of 2% Nital for 40s–60s. In addition, the SEM was equipped with an Energy Dispersive X-ray (EDX) detector that was used to assess the distribution of the main alloying elements in the built parts.

Solid phase distribution and lattice parameter were measured by XRD using a D500 diffractometer (Siemens, Munich, Germany) with $CoK\alpha$ radiation. Diffraction peaks in the range $2\theta = [25^\circ, 90^\circ]$ were considered for determining the solid phase. The acquisition conditions were step size $\Delta\theta = 0.05^\circ$ and step time $t = 8$ s. Background removal and $K\alpha_2$ radiation stripping were performed before peak indexing using Diffrac. EVA software (Bruker, Billerica, Massachusetts). The lattice parameter was measured from the sixth diffraction peak (corresponding to diffraction plane family {222}) in order to achieve high accuracy. The acquisition conditions were step size $\Delta\theta = 0.02^\circ$ and step time $t = 30$ s.

For the analysis of the crystallographic texture intensity using EBSD, a Nova 600 Nanolab Dualbeam FIB/FEG-SEM system (FEI, Hillsboro, Oregon) was employed. EBSD maps and pole figures were

Table 1
Alloying elements concentration range of the employed high-silicon steel powders according to supplier LPW Technologies Ltd.

	Element				
	Si	Mn	C	P	O
Concentration (%wt.)	6.9	0.05	0.01	0.009	0.0065

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