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RESEARCH PAPER

ADDITIVE MANUFACTURING: A NEW CONCEPT FOR END USERS. THE CASE OF MAGNETIC MATERIALS

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ABSTRACT

Metal additive manufacturing (AM) technology is growing up as a technology. Although up today slower and less reliable than traditional production methods, AM systems are showing to be very successful when producing parts with unconventional topologies or in small quantities. In addition, it is showing its capability to produce components with chemical compositions which should not be realized with standard production processes. In this paper some examples are reported of magnetic materials specifically designed for AM. In this work, powder of FeSi electric steel, with 6.5 wt.% Si content is considered to produce samples by AM. Aim of this paper is to investigate the microstructural and texture evolution of FeSi steels, with 6.5% Si, following annealing heat treatment, with the aim of identifying the conditions under which it could be possible to obtain the best magnetization behavior of the alloys.

Keywords: additive manufacturing, magnetic materials, FeSi

INTRODUCTION

The idea of adding material layer by layer during the manufacturing process aimed to produce functional tools and components is perhaps as old as civilization itself as exemplified by traditional assembly methods (**Figure 1**).

Fig. 1 One of the oldest assembly in civilization [1].

Additive manufacturing differentiates from these time-worn techniques specially by the increment scale and spatial accuracy of the added material. Such parameters, for modern systems, are usually in the range of 25–500 µm. The earliest description of a modern metallic 3D printers is reported in a 1972 patent, introducing the concept of "*metal layer fabrication by selectively melting powders using electron, laser, or plasma beams*" [2]. In 1979, a powder laser sintering manufacturing system was described by Housholder [3]. Repeatable accuracy on this scale cannot be achieved without fully digitalized production systems: this is why AM history is relatively short and is generally included in the framework of what is commonly called Industry 4.0. The other factor which strongly limited the development of metal AM is the availability of metallic alloys able to be processed by such process. Metals for AM processing must meet some general requirements. They need to be in a powder, wire, or sheet form and must be machined in a way compatible with their physical properties (for example exposable to air, electrically conductive, etc.). A broad range of candidate materials face with such requirements, although current AM hardware makes the fabrication of parts from oxidation-prone materials difficult (powder is frequently loaded in open air) [4]. There are up today a limited number of commercially available alloys for AM. This is why there remains tremendous opportunity in processing new material and in developing new alloys specifically for AM [5] also with reduced environment impact in the framework of circularity [6]. Recent developments concern the manufacturing of austenitic stainless steels with increased nitrogen content [7-14]. The development of austenitic stainless-steel powders with increased nitrogen content for laser additive manufacturing received great interest since nitrogen is used in this case as alloying element, substituting the expensive and allergenic element nickel, also improving mechanical properties and corrosion resistance. Such alloys cannot be produced by standard casting process due the low nitrogen solubility even if high Mn content is added and is therefore specifically designed for AM [15,16]. Another class of materials which showed to be good candidate to be processed by AM is that of maraging steels (*e.g.*, [17-21]). In such class of materials, the low carbon content and good ductility help to prevent crack formation during rapid cooling [22] typical of AM technologies and no special care are needed to avoid carbides or carbon segregation related problems [22]. Moreover, due to high cost, the maraging steels are often used in the sector, such as aerospace or tool-manufacturing industries, which require the combination of complex geometries and excellent mechanical properties that can be well fulfilled by the AM process [24]. Another important class of materials which are receiving great attention in the framework of AM are Fe-Si steels. Such materials, with Si content ranging 2-7% wt. show excellent electromagnetic properties which increase with increasing Si content which make them candidates for ferromagnetic cores [25-26]. The commonly adopted process to manufacture ferromagnetic cores consists of stacking thin sheets of FeSi steel, coated with a dielectric material [27]. The strategy based on the use of 0.2–0.5 mm thick and coated sheets allow to interrupt the induced currents' circulation path and reduces eddy current losses [28]. Based on this approach, good magnetic properties are conferred to the components. At the same time, this approach provides technological limits. As a matter of fact, it is well known that FeSi steel with 6.5 wt.% Si offers the best soft magnetic properties [29] in terms of high magnetic saturation, low magneto-crystalline anisotropy, low magnetostriction, and high electrical resistivity [30]. Nonetheless commercially, steels with Si content below 3.5–4.0 wt.% are usually preferred since high silicon steels (Si content ≥4.5%) are intrinsically brittle to following their poor workability which does not allow the production of thin sheets through the rolling process [31,32]. The adoption of AM allows to bypass the cold rolling step of traditional route, directly printing the properly designed ferromagnetic core. It has been reported that steels with 6.5 wt.% Si produced by AM appear to be quite promising since in the as-printed condition they exhibit fewer eddy current effects than steels with 3.0 wt.% Si, resulting in greater magnetizing capability and reduced power losses over 50%. Also, the effect of geometry in manufacturing ferromagnetic cores has been investigated and reported [33]. Moreover, it is well-known that the microstructural and texture evolution, induced by the heat treatment, strongly affects the magnetic performances of soft magnetic materials [34].

Aim of this paper is to investigate the microstructural and texture evolution of FeSi steels, with 6.5% Si, following annealing heat treatment, with the aim of identifying the conditions under which it could be possible to obtain the best magnetization behavior of the alloys. In particular, the effect of the annealing heat treatment on the microstructural variation of the FeSi steels additively manufactured by Direct Melting Laser Sintering technology, is reported. The microstructural evolutions, after the heat treatments, are compared with the as-built (untreated) samples.

MATERIAL AND METHODS

In this work, powder of FeSi electric steel, with 6.5 wt.% Si content is considered to produce samples by additive technology. The powder weas produced by gas-authorization process. The characterization of the powder was carried out using a high-resolution scanning electron microscope (FE-SEM Zeiss, Gemini Supra 25) and, as shown in **Figure 2**, the particles morphology is mainly spherical with some satellites and with an elliptical value close to 1. Instead, as regards the average size, the powders have a measurement of about 25 μm.

A system with Laser-Powder Bed Fusion L-PBF technology (EOS – M290) was used to process the powders and to produce the test samples. Five small sample (5 mm x 5 mm x 10 mm) were manufactured, with the long side along the built direction (BD) of the sample. The machine used for manufacturing is equipped with a Yb fiber laser with a nominal diameter of 100 mm and the platform temperature was kept at 200 °C. The process was carried out under an Argon atmosphere with oxygen content below 0.4 %. The optimal printing parameters in terms of specific laser energy E (Jm^{-1}), scan speed v (ms^{-1}), laser power P (W)) were chosen according to [34]. Following the manufacturing process of FeSi6.5 steel, the samples were subjected to annealing heat treatments, in a muffle oven (FM77H), at four different temperatures (900 °C, 1000 °C, 1100 °C and 1150 °C) and a soaking time of 1 hour.

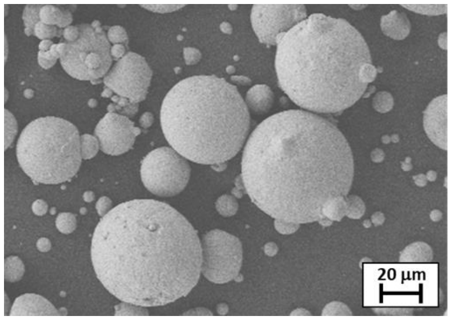


Fig. 2 Powders morphology of FeSi6.5, SEM images.

The test samples were machined along a plane parallel to the BD, polished and etched with 5% Nital solution for 20-40 seconds. The microstructure was then analyzed using an optical microscope (OM) (Eclipse LV150 NL, Nikon, Tokyo, Japan) and an image analysis, to determine the frequency distribution and the average grain size, was performed using dedicated software (AlexaSoft, X-Plus, serial number: 6308919690486393, Florence, Italy). In particular, the microstructural analysis and the measurement of the average grain size were carried out for each state of the steels: as-built samples (untreated) and treated samples, in order to investigate the microstructural evolution after the annealing treatment. Hardness value was measured using a Vickers durometer (HV-50, Remet, Bologna, Italy) with a load of 10 kg (HV_{10}). Three indentations were carried out for each specimen and the average value was considered. Because the structure plays a crucial role in the magnetic behavior of the FeSi alloys, for selected cases, the samples were examined by X-ray diffraction (XRD) (PW 1729, Philips, Eindhoven, The Netherlands). The XRD measurement was performed on the same surface parallel to the built direction and XRD spectra were collected using Mo-K radiation (λ = 0.15408 nm) in the 2Q angle range 15–55 degrees (0.05 degree step scan mode and counting time per step of 5 s). The texture was evaluated by the comparison of peak intensity of each sample with those of a randomly oriented Fe sample taken from the database JCPDS-X-ray-File 6-696 [35].

RESULTS AND DISCUSSION

The solidification structure of FeSi6.5 steel in the as-built (untreated) state along the BD is reported in **Figure 3**. Microstructure appears to be fully columnar with grains directed parallel to the BD as expected for FeSi steels produced by AM technology [28]. In fact, for each layer, the growth of the solidification grains is epitaxial from the underlying layer already solidified. After the annealing heat treatments, the microstructure changes and the grain growth phenomena occur. The microstructure evolution of FeSi6.5 steel at the considered annealing temperatures is reported in **Figure 4**. Just as an example the lower and upper limit temperatures (900 °C and 1150 °C) are reported. Moreover, Figures 4 shows also the frequency of grain size distributions, obtained through image analysis that obey the lognormal function (black line in Figure 4).

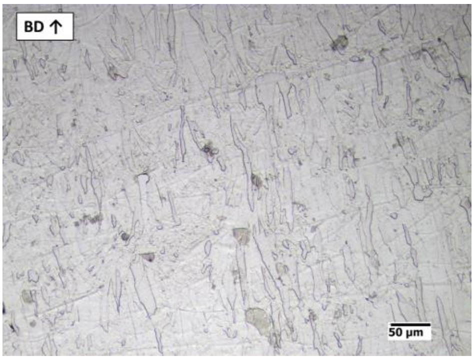
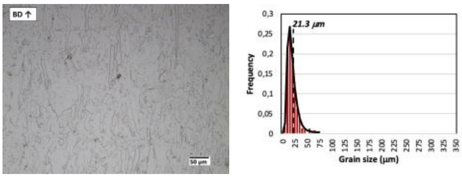
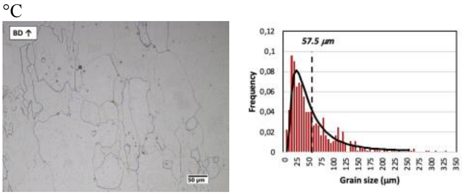


Fig. 3 Columnar microstructure of the section along the built direction (BD) of FeSi6.5 steels in the untreated state (average grain size: 20.1 ± 12.6 µm).



Annealing temperature: 900 °C



Annealing temperature: 1150 °C

Fig. 4 Microstructure evolution of FeSi6.5 steel after annealing heat treatments with different temperatures: (a) 900 °C, (b) 1000 °C, (c), 1100 °C, (d) 1150°C.

The grain growth phenomenon is quite evident: starting from the untreated condition with columnar grain (average grain size of 20.1 ± 12.6 µm), after the annealing heat treatment the microstructure evolves from columnar to equiassic and at 1150 °C and the average grain size is about 57. 5 ± 53.6 µm. Results show that the distribution tends to undergo a slight broadening as the heat treatment temperature increases and also some grains with high dimension (over 250 µm) appear, indicative that the phenomenon of abnormal grain growth is active [36]. The hardness and grain size dependence on the annealing temperature is reported in **Figure 5** showing and evident heat treatment effect on both properties, differently of what it is reported for 3 wt.% Si steels [21]. Therefore, heat treatments after 3D printing appears to have a role in microstructural evolution and hardness behavior in 6.5 wt % steels.

XRD measurements have been carried out to evaluate the texture evolution from the as-built (untreated) sample and the sample heat treated at 1150 °C for one hour. Results are reported in Table 1 and show that in the case of untreated FeSi6.5, the texture is cubic <001> and becomes <110> Goss after annealing heat treatment at 1150 °C for 1 hour. The possible mechanisms underlaying the texture evolution following heat treatment may be found in some heterogenous properties in the as-built sample (*e.g.*, elongated grains, plastic strain due to the thermal stress, etc.).

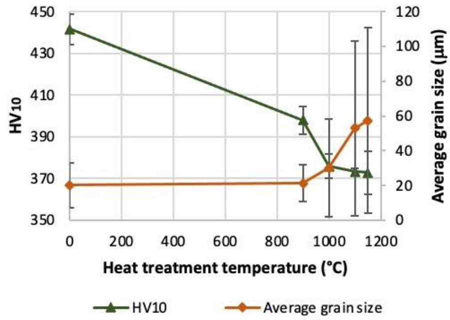


Fig. 5 Hardness values and average grain size evolution of FeSi6.5 steel, following annealing treatment.

Table 1. Intensity of the XRD peaks of FeSi6.5 steel samples, in an untreated state and after heat treatment at 1150 °C for 1 hour. Peak intensities are normalized to the most intense one of each XRD pattern (I = 100). The intensities of an Fe sample with randomly orientated grains (JCPDS X-ray database-file 6-696 [27]) are reported for estimating the texture of the examined samples.

Steel	Normalized Intensity of {h k l} Peaks					
	{110}	{200}	{211}	{220}	{310}	Texture
JCPDS (6-696)	100	20	30	10	12	Random
FeSi6.5 untreated	100	38	25	10	19	<001>
FeSi6.5 / 1150 °C for 1 h	100	8	13	37	10	<110>

CONCLUSIONS

In this work, the microstructural and texture evolution of 6.5 wt.% Si steel, produced by L-PBF technology, following annealing heat treatment is reported. Heat treatments were performed with four different temperatures, in the range between 900 and 1150 °C with a soaking time of one hour. Moreover, the microstructural evolution after the heat treatment was compared with the condition of the steel in the untreated state. The conclusion of the investigation conducted can be summarized as follows:

- in the as-built (untreated) state, the steel produced by L-PBF technology, show a fully columnar solidification microstructure along the built direction, due to the epitaxial growth starting from the layers of material already solidified;
- a significantly visible heat treatment effect is reported in terms of grain size, hardness and texture evolution: the 6.5 wt.% Si steel appear therefore being a “new material” for end user which just should be manufactured by AM technology: its response to heat treatments makes it suitable for properties optimization after being printed.

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