Influence of powder characteristics on final properties of powder-bed laser additively manufactured ods fe-14cr steel

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INFLUENCE OF POWDER CHARACTERISTICS ON FINAL PROPERTIES OF POWDER-BED LASER ADDITIVELY MANUFACTURED ODS Fe-14Cr STEEL

Abstract

Additive manufacturing processes are promising technologies, currently considered as new alternative processing routes for metal components, especially in aerospace, automotive, medical and energy industries. In order to assess the potentialities of additive manufacturing in nuclear industry, ODS (Oxide Dispersion Strengthened) Fe-14Cr steels are produced by selective laser melting (SLM). ODS steels are studied due to their improved resistance under neutron irradiation thanks to a fine dispersion of nanosized Y-Ti-O precipitates. Such materials are produced by a first step of mechanical alloying. The resulting powder has a non-spherical shape and is coarser than powders typically used in SLM equipment. The analyzes such as composition, density, particles size distribution, flowability and morphology are performed on this powder. The milled powder is then used to produce ODS steel parts as raw material or after some modifications such as sieving. The objective of this work is to study the impact of the powder characteristics on the final material properties. As expected, powder characteristics strongly influence the final density of densified parts. The choice of the thickness layer is also an important parameter that needs to be correlated with the particle size distribution of the powder.

Keywords

Additive Manufacturing, Selective Laser Melting, Oxide Dispersion Strengthened Alloys, Powder Characteristics, Process Parameters

1. Introduction

Oxide dispersion strengthened (ODS) ferritic steels are currently produced by powder metallurgical process. Mechanical alloying (MA) consists of milling steel powder with Y_2O_3 and TiH_2 powdersto distribute homogenously Ti and Y atoms inside the ferritic matrix. The powder is then consolidated by hot extrusion or hot isostatic pressing to obtain a bulk material. This step allows the precipitation of Y-Ti-O oxides inside the matrix, which lead to an improvement in mechanical and physical properties, especially for high temperature applications. Considering the limitations regarding the final shape complexity of components obtained by these traditional fabrication routes, the evaluation and development of alternative production methods are currently studied in order to increase the widespread use of ODS alloys.

In the frame of assessing the potentialities of additive manufacturing to manufacture ODS complex parts, a Fe14Cr1W + 0.3% Y_2O_3 + 0.3% TiH_2 milled powder is consolidated by

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Selective Laser Melting (SLM). This process depends on a wide range of parameters such as laser power, scanning speed, hatch distance, scanning strategy… An optimization of these parameters is performed to get dense SLM builds. These processing parameters are not the only parameters to take into account to increase the density. This study aims at investigating the influence of the powder characteristics on the properties of final parts.

Non-spherical ODS Fe-14Cr milled powder and standard spherical atomized Fe-14Cr powder are used and characterized for comparison. Observations and measurements such as chemical composition, density, particles size distribution, flowability and morphology are performed on these powders before consolidation by SLM process.

2. Materials and Methods

A Fe-14Cr-1W-0.3Mn-0.3Si-0.2Ni steel powder (supply by Nanoval) was milled with 0.3%wt. of Y_2O_3 oxide powder and 0.3%wt. of TiH_2 hydride powder during 176 hours under hydrogen atmosphere by Mecachrome on MATPERF Platform. The integration of these compounds, Y_2O_3 and TiH_2, aims at the formation of stable oxide particles such as $Y_2Ti_2O_7$ or Y_2TiO_5 in the stainless steel matrix [1,2].

The gas atomized powder from Nanoval will be called “Powder A” and “Powder A30” when only the finest particles inferior to 30 µm are kept after sieving. The milled powder will be called “Powder M” and “Powder M80” when this powder is sieved at 80 µm.

Consolidations of powders are conducted on a TruPrint series 1000 SLM machine (TRUMPF GmbH), equipped with a 200 W Yb-fiber laser (λ=1.064 µm). The consolidation process is performed under argon atmosphere in a sealed chamber. An argon gas injection ensures a constant flow in the build chamber with an imposed oxygen concentration lower than 100 ppm during the process. All samples consolidated by SLM process are cubes of 10 mm side. Individual layers of these samples are scanned with parallel lines using bidirectional vectors separated by a specific hatch distance (HD). The scanning direction is altered by 90° between two consecutive layers, as shown in Fig. 1. Samples densities are measured following the Archimedes method.

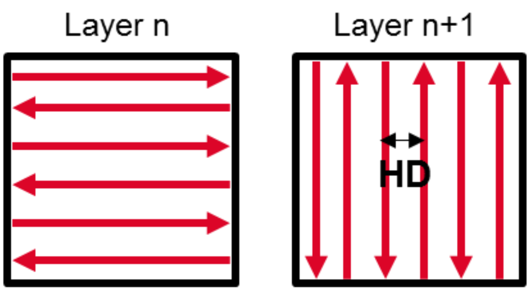

Fig. 1: Schematic of scanning strategy using parallel lines rotated of 90° between each layer (HD: hatch distance)

3. Powder’s characterisation and comparison

Fig. 2 shows SEM images of Powder A and Powder M. Observations show that the atomized powder is composed by spherical particles, contrary to the milled powder.

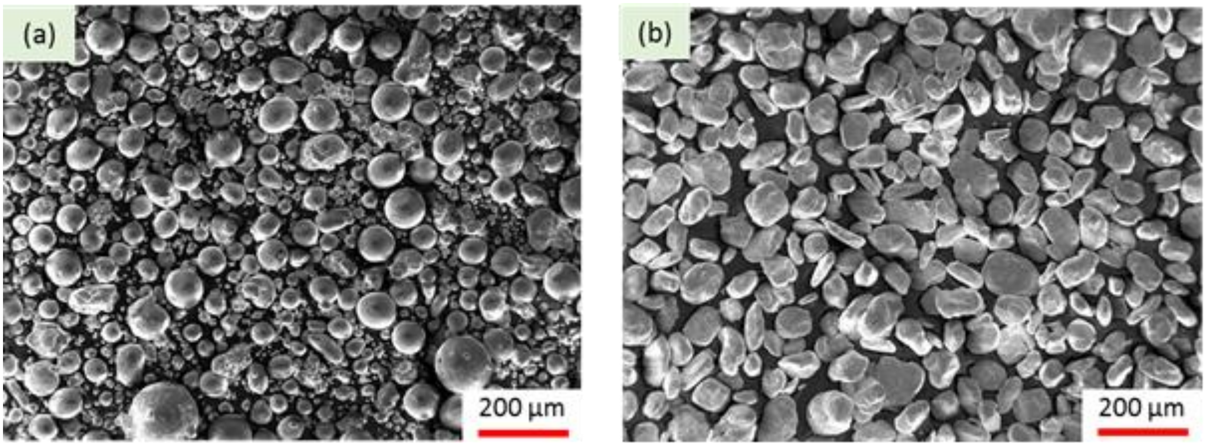

Fig. 2: SEM images of a) as-received Fe-14Cr stainless steel atomized powder (Powder A) and b) as-received ODS milled powder (Powder M)

McGeary shows that spherical particles of one size pack according to an orthorhombic pattern with about 62.5% of theoretical density [3]. Smaller are the particles, harder is the packing procedure. It is necessary to have powder with suitable size ratio to improve the density of powder layer. Indeed, fine particles can fill voids left between coarse particles, as shown by Karapatis et al. [4].

Particles size and flowability of the studied powders are measured and results are presented in table 1. The theoretical densities are measured thanks to a helium pycnometer. The measurement of apparent density is described in ASTM B212 [5]. Tapped density is quantified with a Densitap ETD-20 from Granuloshop®, which follows the USP standard. Particles sizes are measured thanks to a granulometer Partica LA-950 from Horiba®.

Table 1 : Comparison of powders' characteristics

Powder	Density (g/cm^3)	Tapped density (g/cm^3)	Apparent density (g/cm^3)	Hausner ratio	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
A	7.938 ± 0.003	4.9	4.3	1.14	31.5	53.3	94.2
A30	/	5.1	4.2	1.21	9.5	16	25.4
M	7.926 ± 0.007	4.7	3.7	1.27	47.6	68.9	103.0
M80	/	4.3	3.6	1,19	45,2	61.8	86.4

Powder A is finer and more spherical than Powder M. This difference in shape and size probably explains the best tapped and apparent densities in comparison with Powder M. It is easier to pack spherical particles rather than non-spherical particles. Sieving the Powder M allows to remove the coarsest particles and decrease the average particle size (d_{50}) of about 7 µm.

Fig. 3 shows a SEM image of Powder M80. The sieving allows to obtain powders with a narrow size range and homogeneous shape. However, this lower size distribution leads to a decrease in the tapped density of the powder. Powder A30 contains only smaller particles with a size inferior to 30 µm. In spite of the narrow size distribution of Powder A30, the tapped and apparent densities are relatively high. These both parameters seem to be not influenced by the smaller size of powder particles contrary to the observations of McGeary and Karapatis [3,4]. The size distribution must be enough to get a good packing of this powder.

Hausner ratio is linked to the flowability. This parameter is defined as the ratio between tapped and apparent density. Abdullah et al. reports that powders with Hausner ratio inferior to 1.25 are considered as powder with good flowability and powders with Hausner ratio superior to 1.4 are considered as cohesive powder [6]. All the powders used in this study display good flowability with regard to the requirements for SLM. Powder A30 is not cohesive and can be efficiently packed.

A scraper spreads the powder during the process. The powder bed density estimation is a compromise between tapped and apparent density, due to the effect of the scraper on the powder. The powder bed density is probably closer to apparent density rather than tapped density. Best densities can be expected for SLM parts built with powders A and A30 since these powders have better apparent density than milled powders [7].

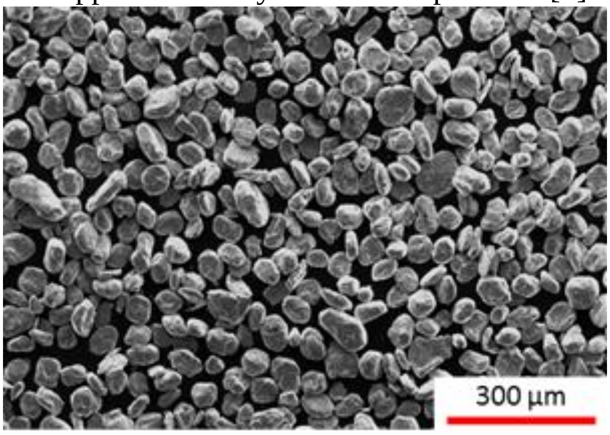

Fig. 3: SEM image of milled powder sieved at 80 µm (powder M80)

4. Influence of powder's characteristics on density of SLM builds

Consolidation by SLM process are performed with all the different powders following the same set of processing parameters. These parameters are fixed thanks to energy density E_d, which can be expressed as follow:

$$E_d = \frac{P_{laser}}{t_{layer}\ x\ HD\ x\ V_{scan}} \quad \text{en J/mm}^3$$

Where P_{laser} is the laser power, t_{layer} is the thickness of layer, HD is the hatch distance and V_{scan} is the scanning speed.

Fig. 4 shows the evolution of the density of consolidated samples as a function of energy density for the parts consolidated with the different powders. It can be seen that the use of atomized powder (A and A30) leads to the increase in the density of the final parts on a wide range of processing parameters: Finer is the powder, wider is this processing parameters range. This phenomenon can be explained by the speed to melt small powder particles. The spherical shape of these powders increases probably the density of powder bed and finally the density of parts as suggested by the apparent and tapped densities. The use of spherical powders also reduces the roughness of samples in comparison with the use of milled powders. Powder's morphology has a strong influence on the properties of the final parts as it was also observed by Spierings et al. [8]. The authors manufacturing SLM samples employing standard IN625 as well as ODS-IN625 powders. Their study show that the processing parameters range is much narrow when ODS-IN625 powder is used due to its irregular shape. The morphology of the particles does not only affect the flowability but also the laser / powder interactions.

The densities of parts built with Powder M80 are higher than those build with Powder M for energy densities inferior to 200 J/mm^3. This phenomenon could be attributed to the higher homogeneity in shape and size for Powder M80. Powder M80 would have higher powder bed density than Powder M, which allows to get continuous tracks during the laser melting. Measurements with high speed camera should be done to verify this theory.

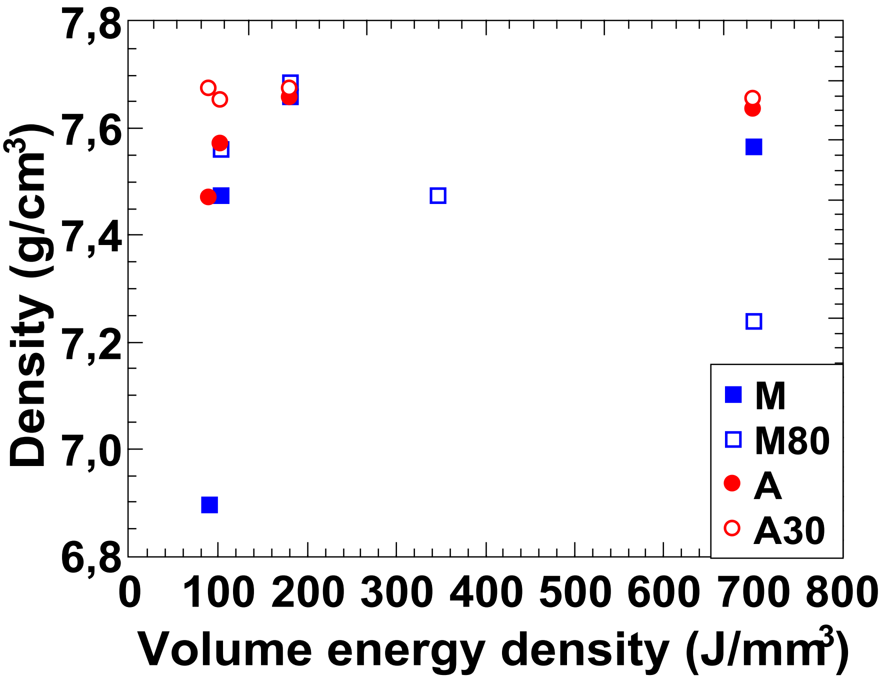

Fig. 4: Evolution of the density consolidated samples as a function of energy density for the different powders

Conclusion

This work shows the elaboration feasibility of dense additively manufactured ODS-Fe14Cr parts. Mechanically alloyed powders can successfully be employed as raw material to perform SLM consolidations. However, the processing parameters range will be narrower with such powders rather than spherical powders. The non-spherical shape of milled powders affects their packing ability, thus decreasing the final parts densities in comparison with atomized powders.

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