

Plasma Spheroidization of SS316L: Preliminary Experimental Results.

P.Iovane, C. Borriello, S. Portofino, L. Tammaro, G. Pandolfi, G. Rametta, S. Galvagno
SSPT-PROMAS-NANO CR. ENEA “Portici”

Introduction

The new frontiers of additive manufacturing (AM) involve several emerging printing techniques that promise to revolutionize the manufacturing processes in many industrial fields. Indeed, the “additive” approach allows to create objects with geometries and shapes unobtainable by using the classic “subtractive” one. This increased attention toward 3D printing techniques boosts the development of new printing materials, from polymers and resins to metals and ceramics. Among these, metal powders occupy a rapidly growing market. The development of spherical powders, with high packing capacity and therefore good flowability, becomes a primary requirement for printing. Usually, these powders are produced by Water Atomization (WA) and Gas Atomization (GA); however new techniques such as Plasma Atomization (PA) and Plasma Spheroidization (PS) are gaining increasing interest. In this frame, ENEA developed a new prototypal plant for the production of powders for AM; the system based on DC thermal plasma technology, was own designed and installed at ENEA Portici Research Centre. This work was carried out on SS316L angular stainless-steel powder with the aim of exploring and identifying the best process parameters for the production of spherical powders.

Experimental

Commercial SS316L powders provided by Thermofisher have been used for the experimental work. The raw powders were processed by a DC thermal plasma plant installed at ENEA Portici RC. Figure 1 shows the flow sheet of the plant. The plasma operates under a light vacuum. The torch is fitted in the upper part of a jacketed-cylindrical stainless steel reactor cooled with circulating cold water. The reactor is equipped with a collection tank, where the produced powders are collected along with the unreacted materials. At the top of the reactor, one nozzle feeds the powder directly into plasma flame. Tests have been conducted using argon (Ar) as the main gas to light the plasma and helium (He) as secondary gases to improve the flame conditions. Argon is also used as carrier for powders injection. Table 1 shows the experimental conditions for SS316L<100 mesh tests; plasma power was varied in the range 12-23 kw, while other parameters were kept constant.

XRD, SEM and Flowability analysis were performed to characterize reagents and products.

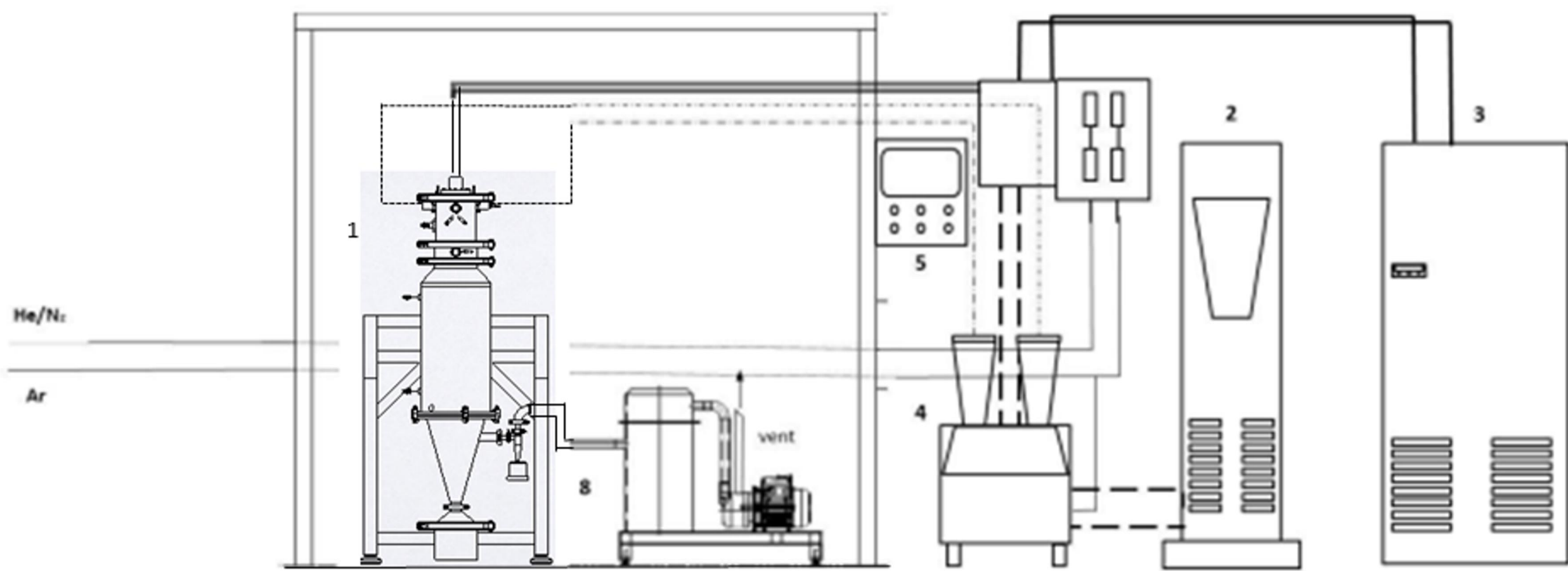


Fig.1 - Plasma plant flow sheet: 1) Plasma Torch; 2) Power Supply; 3) Chiller; 4) Powder Feeder; 5) Control Unit; 6) Reactor; 7) Collection Tank; 8) Bag Filter.

| Test code | Power (kW) | Ar (slm) | He (slm) | Ar carrier (slm) | Pressure Test (kPa) | Feeding rate (g/min) |
|-----------|------------|----------|----------|------------------|---------------------|----------------------|
| SSPL5 | 12,5 | 40 | 10 | 1 | 90 | 5 |
| SSPL6 | 17 | 40 | 10 | 2 | 92 | 5 |
| SSPL7 | 21 | 40 | 10 | 1,5 | 91 | 5 |
| SSPL8 | 21 | 40 | 10 | 1 | 87 | 5 |
| SSPL9 | 21,5 | 40 | 15 | 1 | 105 | 5 |
| SSPL10 | 23 | 50 | 15 | 1 | 92 | 5 |

Tab 1 : experimental conditions for SS316L <100 mesh

Results and Discussion

During the process, injected particles absorb energy by the plasma and if the energy is enough, they melt; out of the plasma, the surface tension forces determine their spherical shape during the cooling phase. If a higher energy is employed, the smallest particles evaporate, resulting in the formation of nanoparticles deposited on the reactor wall and on the biggest particles surface. After processing, all samples present spherical shape with the presence of nanoparticles that increases with plasma power.

SEM analyses of raw material and processed powder, after classification, are reported in figure 2. The starting material shows angular and irregular shape particles with a quite wide diameters distribution, ranging from particles lower than 10 microns, to aggregates larger than 100 microns. Processed powders have regular and spherical shape. The best results were obtained under SSPL9 and 10 conditions. In this case, nearly spherical powders with high circularity (0.93) and improved flowability (24 sec/50 g) were obtained. Table 2 reports a comparison between the results obtained and data available in literature, showing that flowability is in line with the commercial products.

Figure 3 shows XRD characterization. The main phase of all treated powders is the austenitic, similarly to the starting material. The ferritic phase is almost absent; it is detected only in the spectra of the sample obtained at the lowest power used (12 kW). In addition, an increasing formation of oxides with the rising of the plasma power is highlighted. This is probably due to the increasing presence of nanoparticles produced at higher plasma power and more susceptible to oxidation.

Conclusions

The experimental tests conducted on SS316L powder using a DC thermal plasma plant installed at ENEA Portici RC have shown that starting material particle size plays a relevant role in the determination of process conditions. All samples show spherical particles. The best results were obtained under SSPL9 and 10 conditions. The average value of the circularity of the powders, a parameter to measure the degree of spheroidization, resulted in all tests ≥ 0.8 and flowability data around 30 s/50 g. These results are in line with the literature references for powders used for SLS and SLM applications. Further tests are however necessary to better characterize the powders obtained. Besides post-processing powder “classification” (leaching and sieving) is very important to obtain usable products, in analogy with commercial processes and will surely be explored in subsequent works.

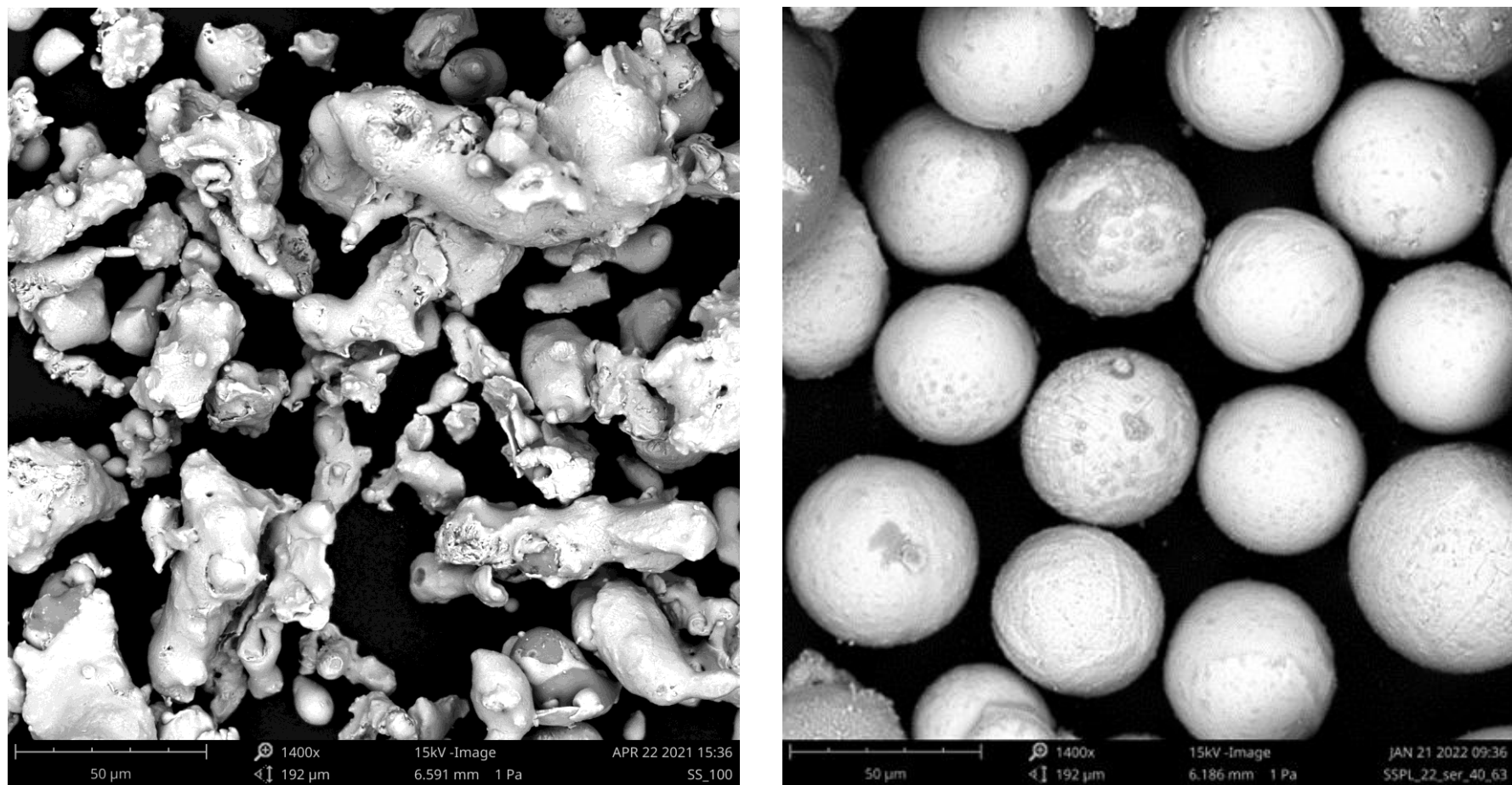


Fig.2 –SEM images on powders before (left) and after (right) plasma treatment (SSPL9 cond.)

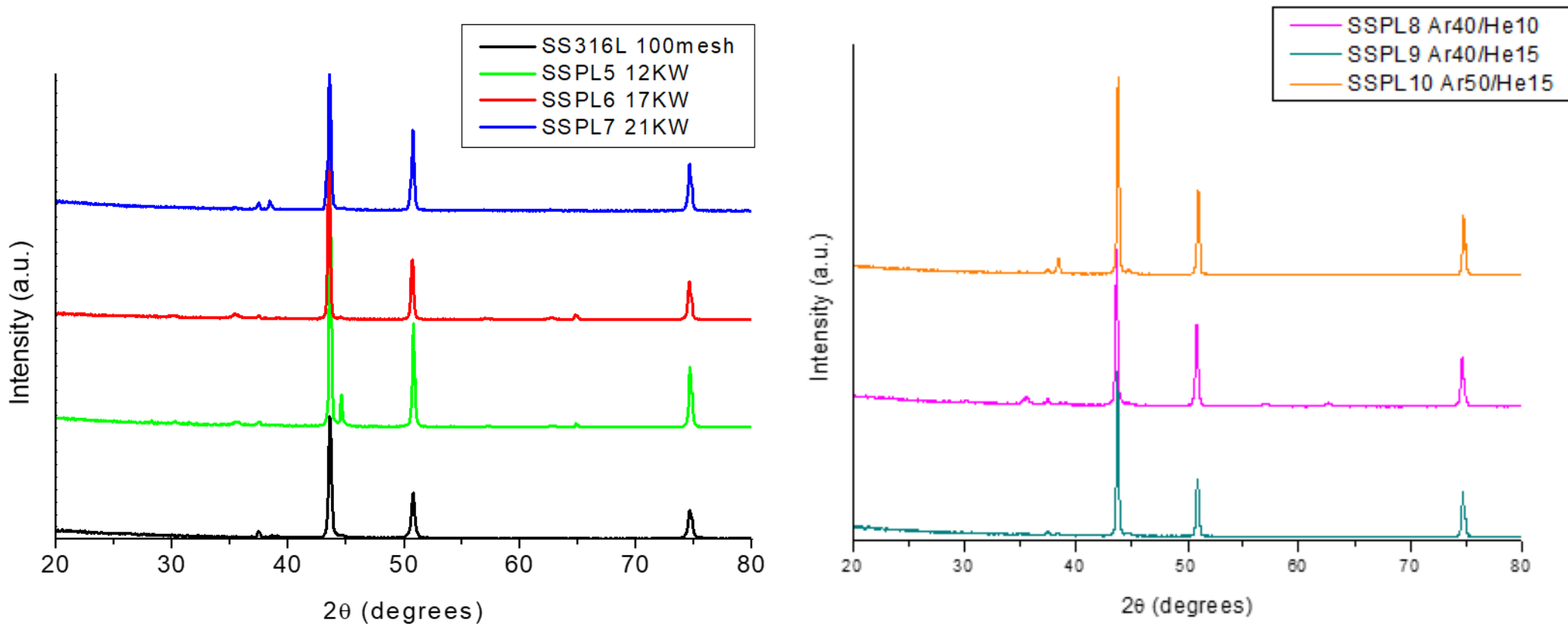


Fig.3 – XRD on powder before and after plasma treatment

| POWDER | PROCESS | FLOWABILITY (s/50g) |
|----------------------|------------------------|---------------------|
| HOGANAS AB | GAS ATOMIZATION | 18,1 |
| HOGANAS AB | WATER ATOMIZATION | 37,4 |
| MIMETE METAL POWDERS | GAS ATOMIZATION | ≤ 30 |
| THIS WORK | PLASMA SPHEROIDIZATION | 24 |

Tab 2: flowability

Acknowledgement

This work has been conducted within the framework of the “PTR2019-2021” Ministero dello Sviluppo Economico- ENEA, Project 1.3 «Materiali di Frontiera per Usi Energetici» and “PTR2022-2024” Ministero dello Sviluppo Economico-ENEA, Project 1.4 «Materiali di Frontiera per Usi Energetici»