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LASER POWDER BED FUSION OF PURE NIOBIUM FOR PARTICLE ACCELERATOR APPLICATIONS*

S. Candela[1,†], M. Bonesso, V. Candela[1], R. Dima, G. Favero[2], A. Pepato, P. Rebesan, M. Romanato,
Istituto Nazionale di Fisica Nucleare (INFN) – Section of Padova, Padova, Italy
E. Chyhyrynets[3], D. Ford, C. Pira, INFN – Legnaro National Laboratories, Legnaro, Italy
[1] also at Center for Fusion Research, University of Padova, Padova, Italy
[2] also at Department of Management and Engineering, University of Padova, Vicenza, Italy
[3] also at Department of Chemical Sciences, University of Padova, Padova, Italy

Abstract

Niobium is particularly appreciated for its superconductive properties. One of the main applications of this metal in Nuclear Physics is the production of superconducting radiofrequency (SRF) cavities for particle accelerators. Additive manufacturing (AM) gives the chance to fabricate objects with very complex shapes; also, high melting temperature and hard-to-machine materials can be easily processed. However, AM is not free from challenges, and the creation of devices such as the SRF cavities is not trivial. In this work, the characterization of pure niobium produced by laser powder bed fusion (LPBF) and a fine-tuning of the printing parameters have been carried out. Much emphasis was put on the development of innovative contactless supporting structures for improving the quality of downward-facing surfaces with very small inclination angles. A relative density higher than 99.8% was achieved and the efficiency of such innovative supports was demonstrated, as they made the fabrication of seamless SRF cavities possible. Smoothing surface treatments and performance tests on additively manufactured cavities were also performed.

INTRODUCTION

A superconducting resonant cavity is an energy storage device employed for the radiofrequency and microwave range [1]. It provides energy to charged particles in particle accelerators. In order to accelerate particles, an alternating electric field must be employed. It follows that an alternating magnetic field is generated at the walls of the cavity, and surface shielding currents provoke the so-called skin effect: the material of the cavity sees localized heating on its surfaces by the Joule effect due to the accumulation of these currents in those regions. Indeed, superconducting materials are the most suitable materials for this application, because of the intrinsic characteristic of superconductivity: they show extremely low surface resistivities when temperature undergoes their critical temperature T_C, so the energy losses are drastically reduced.

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† silvia.candela@pd.infn.it

The subtractive manufacturing processes traditionally employed for producing RF cavities are often cost-prohibitive for material needed and for the consistent amount of material wasted that is not possible to re-use. The laser powder bed fusion (LPBF) process does not see this problem because the unmelted particles of metal powder can be easily recycled. This is a huge advantage if an expensive metal like niobium must be processed. Additive manufacturing (AM) allows to the creation of components with highly complex shapes in a very reduced time, but some drawbacks of these techniques are evident (i.e., low surface finishing and residual porosity) and have a great impact on the performances of the manufactured parts. However, some drawbacks can be present, like the poor surface quality of the parts produced, the residual porosity, and impurities of the raw materials used [2].

In the LPBF process, a powerful laser beam selectively melts the feedstock powder. The powder bed is spread by a blade onto a metallic building platform, after the exposure phase, the platform is moved down and a new layer of powder is formed again. The layer thickness is one of the several parameters that influence the properties of the objects produced with this process. The choice of its value is dictated mostly by the particle size distribution of the powder employed.

Firstly, a parameters fine-tuning campaign was performed to individuate pure niobium's optimum process window. The as-built surface quality of the AM parts was then evaluated. Indeed, the high surface roughness of the objects produced with AM techniques has always represented a problem. Concerning LPBF, many factors play a role, like the already mentioned layer-thickness, laser exposure parameters (contour, down skin, up skin are the most relevant), geometry of the parts, and so on. More information about this can be found in references [3, 4]. In this experience, the minimum self-supporting angle of niobium was determined and the down skin parameters were extensively studied. Innovative contact-free supporting structures were also developed in order to improve the quality of the as-built downward-facing surfaces. These new supports consist of additional parts printed under the objects, with the up skin surfaces of the former parallel to the downward surfaces of the latter and without any connection between the two. In principle, such supporting structures act as heat sinks, enhancing the heat dissipation from the principal parts and, because of this, the down skin quality improves considerably [5].

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Finally, 6 GHz pure niobium resonant cavities were produced via LPBF, and their performances were investigated.

MATERIALS AND METHODS

For this experience, the AMtrinsic® niobium spherical powder produced by Taniobis GmbH (Goslar, Germany) was used, with a PSD ranging from 18 µm (D10) to 63 µm (D90). The layer thickness was set equal to 30 µm. The machine that was employed is an EOS M100 DMLS (Electro-Optical System GmbH, Krailling, Germany), which is equipped with a ytterbium-YAG red laser. The maximum power that can be reached is 200 W. The process is carried out in a controlled environment: Argon is the inert gas used to minimize the oxygen level inside the printing chamber. The oxygen content was kept below 0.15%.

Different combinations of hatch spacing, laser power, and scanning speed were employed to produce cubic samples for density estimation. The relative density was then measured according to the Archimedes method: the mass of each sample was measured several times in air first, and then in distilled water. The density of the additively manufactured material was determined according to the following expression:

$$\rho_{AM} = \frac{m_{air}}{m_{air} - m_{wet}} \cdot (\rho_{H2O} - c) + c \qquad (1)$$

where m_{air} is the average mass of the sample in air, m_{wet} is the average mass measured dipping it in water, ρ_{H2O} is the density of water as a function of room temperature, and c is a corrective term related to the effect of air, which is equal to 0.0012 g/cm^3.

A set of overhang samples were then produced to investigate the minimum self-supporting angle of additively manufactured niobium parts. Angles from 50° to 20° were considered. At the same time, the down skin parameters and the effectiveness of the contact-free supporting structures were investigated. For the down skin exposure parameters, several power and scanning speed values were tested, while different gap widths were examined. The as-built surface quality was finally evaluated by measuring the surface roughness, according to the ISO 25178 standard. A Sensofar S Neox (Sensofar, Barcelona, Spain) optical profilometer was employed for these tests.

Once the density and surface quality were maximized and optimized, respectively, pure niobium 6 GHz cavities were produced. On two of these prototypes (small cavity and big cavity), tightness under vacuum and resonant frequency were tested. Surface treatments were also performed at INFN-LNL. Three vibro tumbling (VT) steps were done on the cavities. A solution of water and soap with ceramic abrasive media was used for all the polishing steps. The treatment strategy is here reported in detail for each cavity.

Small cavity The following VT steps were carried out:

- Step 1 (60 min): Angle cut cylinder + 10 mL of H_2O and soap.
- Step 2 (150 min): Angle cut cylinder + 12 mL of H_2O and soap.
- Step 3 (180 min): Angle cut tristar + 12 mL of H_2O and soap.

Big cavity For this prototype, the VT steps are slightly different:

- Step 1 (180 min): Angle cut cylinder + 10 mL of H_2O and soap.
- Step 2 (300 min): Angle cut cylinder + 12 mL of H_2O and soap.
- Step 3 (24 h): Angle cut cylinder + 12 mL of H_2O and soap.

Critical temperature and Residual Resistance Ratio (RRR) were also measured to evaluate the quality of the material processed via LPBF for superconducting applications. The critical temperature T_C was measured using an inductive method, exploiting the Meissner-Ochsenfeld effect. The RRR was then evaluated by measuring the resistivity at 300 K and 10 K, respectively:

$$RRR = \frac{\rho_{300\,K}}{\rho_{10\,K}}. \qquad (2)$$

For these tests, small samples of additively manufactured niobium were also produced. The samples were tested in as-built conditions. For avoiding microstructural modifications due to post-process treatments, like the removal with mechanical tools or Electron Discharge Machining (EDM) of the parts from the platform, the specimens were built on supports easy to break manually, so without the risk of causing localized heating.

RESULTS AND DISCUSSION

The maximum density obtained from the samples produced is above 98.7%

The minimum overhang angle that was achieved without any kind of support was 35°, while the use of a contact-free supporting system allowed printing successfully downward facing surfaces characterized by inclination angles smaller than 35°. For the samples produced setting a wider gap between the upper part and support, however, the smallest angles tested failed.

Surface roughness measurements were performed on the down-skin regions of samples without supports and contactless supports. It was proven that the use of such supports can improve the as-built surface finish: from a radium of 65 µm obtained measuring a non-supported sample, the surface roughness was lowered to 35 µm setting a proper gap of the contact-free supports.

Prototypes of pure Niobium 6 GHz cavities were then printed and preliminary tests were performed. Figure 1 shows the seamless cavities successfully produced. The parts were produced in different printings, because of the small printing volume available on EOS M100. The first prototype cut-offs were shortened with respect to the standard length.

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Figure 1: Seamless niobium cavities produced with LPBF process.

Tomography analyses were carried out in order to control the wall thickness and the quality of the internal surfaces. Despite the difficulties due to the high Z of niobium, it was possible to see that the material printed is very massive: no internal porosity was detected, and the wall thickness was homogeneous and constant, while some irregularities were visible in correspondence of the upper iris of the cavities: dross formation on the internal surfaces was noticed in the connection of the cell with the cut-off, as can be seen in Fig. 2. This is probably due to poor heat exchange at that height of the job during the printing process.

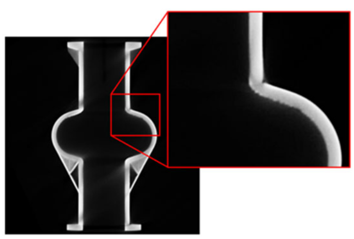

Figure 2: X-ray tomography of the big prototype.

An internal inspection was also performed, from which the as-built surface finish was revealed to be highly uniform, thanks to the use of an internal contact-free supporting structure. This is a very important aspect that will help in obtaining a good quality of the internal surfaces after the smoothing surface treatments, and thus, good performances of such cavities.

The leak tests were also successfully passed: no leakages were detected in any prototype produced and a vacuum level equal to 10^{-12} mbar.

The resonance frequency was tested in the as-built conditions at room temperature. The measurements revealed that the LPBF process guarantees great reproducibility of the performances of the components produced: the frequency values obtained are very close to the ideal one (6 GHz). On the small cavity, a RF of 5.999 GHz was measured, while 6.027 GHz was the RF of the big cavity.

After the VT surface treatments, the internal surfaces of the prototypes became very smooth, despite some pitting-like defects are still visible. Anyway, no new cracks or leakages were detected after performing the VT process. Figure 3 shows the pictures of the internal inspections performed before (as-built conditions) and after the VT steps. The surface roughness was not measured since it is not possible to access the cavity with a probe without cutting the prototype. Only visual inspections were carried out so far. However, the improvements made by VT are visible.

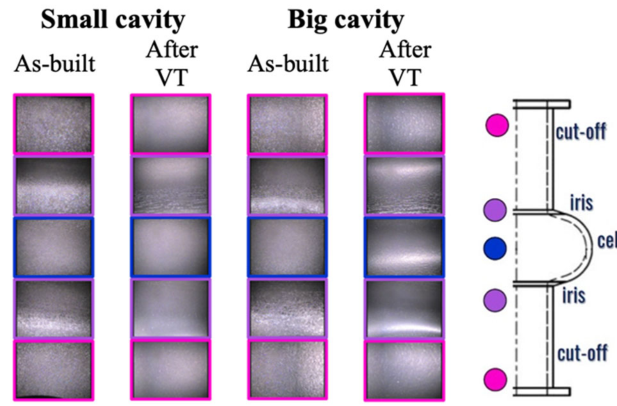

Figure 3: Internal inspection of the small prototype.

From the measurements performed on the samples of additively manufactured niobium, the critical temperature T_C obtained is equal to 9.15 ± 0.01 K. The RRR value determined is 8. These results are in accordance with those obtained with conventionally processed niobium.

CONCLUSIONS

In this study, the LPBF process of pure niobium was investigated. A process parameters fine-tuning campaign was carried out in order to maximize the density and optimize the as-built surface finish. In particular, the down skin parameters were evaluated in deep. The production of seamless pure niobium 6 GHz accelerating cavities with the LPBF AM technique was then successfully performed. Performance tests were also executed on the additively manufactured parts to determine the effectiveness of such production method. Very promising results have been obtained. Additional surface treatments can be performed to further reduce the surface roughness and eliminate the point-like defects previously mentioned. The research is ongoing.

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