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ADDITIVE MANUFACTURING OF PURE NIOBIUM AND COPPER USING LASER POWDER BED FUSION FOR PARTICLE ACCELERATOR APPLICATIONS*

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Abstract

Additive Manufacturing (AM) provides a unique opportunity to produce objects with complex shapes and enables the processing of materials with high melting points and difficulty in machining. This study focuses on the characterization of pure niobium and copper 6 GHz cavities fabricated using Laser Powder Bed Fusion (LPBF) and the optimization of printing parameters. Special attention was given to the development of innovative contactless supporting structures that enhance the quality of downward-facing surfaces with extremely small inclination angles. Through these efforts, a relative density exceeding 99.8% was achieved, demonstrating the effectiveness of the novel supports in enabling the seamless fabrication of SRF cavities. Additionally, surface smoothing treatments were applied, and performance tests were conducted on the additively manufactured cavities.

INTRODUCTION

The traditional subtractive manufacturing processes used to produce RF cavities are often costly due to material requirements and the inability to reuse wasted material. However, Laser Powder Bed Fusion (LPBF) overcomes these issues as the unused metal powder particles can be easily recycled, making it advantageous for processing expensive metals like niobium. Additive Manufacturing allows for the creation of components with complex shapes in a short time. Nonetheless, there are drawbacks associated with these techniques, such as low surface finishing, residual porosity, and impurities in the raw materials used.

In the LPBF process, a powerful laser selectively melts the feedstock powder, which is spread onto a metallic platform. The platform is then lowered, and a new layer of powder is formed. The choice of layer thickness is influenced by factors like Particle Size Distribution of the powder.

To optimize the process, a parameters fine-tuning campaign was conducted to identify the optimum process window for pure niobium. The surface quality of the additively manufactured parts was evaluated, considering the challenge of high surface roughness associated with additive manufacturing techniques. [1, 2] The minimum self-supporting angle of Nb was determined, and the down skin parameters were extensively studied. Additionally, innovative contact-free supporting structures were developed to enhance the quality of the downward-facing surfaces. These supports act as heat sinks, improving heat dissipation and significantly enhancing the quality of the downward surfaces. [3] A total of three Nb cavities were manufactured for this work.

Two pure copper cavities were also produced to test the effectiveness of surface treatments. The future development involves printing 6 GHz copper cavities, performing surface treatments on them, and applying superconducting coatings. The ease and speed of creating these cavities, once the printing processes are optimized, make this alternative highly attractive compared to other, more conventional, manufacturing methods.

EXPERIMENTALS

Niobium Cavities

In this work, AMtrinsic® Niobium spherical powder provided by Taniobis GmbH (Goslar, Germany) was used, with a particle size distribution (PSD) ranging from 18 µm (D10) to 63 µm (D90). The layer thickness was set at 30 µm. The additive manufacturing machine used was an EOS M100 DMLS (Electro-Optical System GmbH, Krailling, Germany), equipped with a Yb:YAG red laser with a maximum power of 200 W. The printing process was conducted in a controlled environment using argon (Ar) as the inert gas to minimize the oxygen level within the printing chamber, maintaining the O_2 content below 0.15%.

To estimate density, cubic samples were produced using different combinations of hatch spacing, laser power, and scanning speed. The relative density of each sample was measured using the Archimedes method, where the mass of each sample was measured multiple times in air and then in distilled water.

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Overhang samples were also produced to investigate the minimum self-supporting angle of additively manufactured niobium parts. Various angles ranging from 50° to 20° were considered. Concurrently, the down skin exposure parameters and the effectiveness of contact-free supporting structures were examined. The down skin exposure parameters involved testing different power and scanning speed values, as well as examining various gap widths. The surface quality of the printed objects was evaluated by measuring surface roughness following the ISO 25178 standard, using a Sensofar S Neox optical profilometer (Sensofar, Barcelona, Spain).

Once the density and surface quality were optimized, pure niobium 6 GHz cavities were manufactured.

To assess the quality of the niobium cavities manufactured via LPBF for superconducting applications, measurements were conducted to determine the critical temperature (Tc) and the Residual Resistance Ratio (RRR). The critical temperature was measured utilizing inductive method and the RRR was evaluated by measuring the resistivity at two temperatures: 300 K (room temperature) and 10 K (low temperature).

To ensure the accuracy of the tests, small samples of additively manufactured niobium were produced specifically for these experiments. The samples were tested in their as-built conditions to avoid any microstructural modifications that may occur during post-process treatments such as removal with mechanical tools or Electron Discharge Machining (EDM). To facilitate easy removal without the risk of localized heating, the specimens were built on supports that could be manually broken. This approach ensured that the test results accurately reflected the properties of the additively manufactured niobium samples without any potential artifacts introduced by post-processing procedures [4].

Copper Cavities

In this work, two 6 GHz cavities were fabricated using commercially pure copper powder. The printing process was conducted using the TRUMPF TruPrint 5000 machine, which is equipped with a green laser operating at a wavelength of 515 nm and a maximum nominal power of 1 kW. The cavities are denoted as T1 and T2 [5].

Vacuum tightness and resonant frequency tests were performed on all cavities, both Nb and Cu.

Surface treatments included: Traditional Mass finishing process (MF), at Rösler Italiana S.r.l., Vibro-Tumbling (VT) and Electropolishing (EP) at INFN-LNL. Tc and RRR measurements were done as well in Legnaro.

RESULTS AND DISCUSSION

The Nb samples produced achieved a maximum density of over 98.7%. Without any support, the minimum achievable overhang angle was 35°. However, with the use of a contact-free supporting system, successful printing of downward-facing surfaces with inclination angles smaller than 35° was possible. It should be noted that samples with wider gaps between the upper part and support experienced failures when attempting to achieve smaller angles.

Surface roughness measurements were conducted on the down-skin regions of samples both with and without contactless supports. The results demonstrated that the use of these supports can enhance the surface finish. The surface roughness decreased from 65 µm (measured on a non-supported sample) to 35 µm by setting an appropriate gap for the contact-free supports.

Subsequently, 3 prototypes of pure Nb 6 GHz cavities were additively manufactured, and preliminary tests were conducted. Figure 1 illustrates the successful production of seamless cavities. Due to the limited printing volume available on the EOS M100 machine, the parts were produced in multiple printing sessions. As a result, the cut-offs of the first prototype ("small cavity") were shortened compared to the standard length.

Figure 1: Niobium AM cavities.

Tomography analyses were conducted on a Nb cavity to monitor the wall thickness and internal surface quality of the printed components. Despite the challenges posed by the high atomic number of niobium, the analyses revealed that the printed material exhibited a solid and dense structure. No internal porosity was detected, and the wall thickness was found to be uniform and consistent. However, some irregularities were observed specifically in the upper iris region of the cavities. Notably, dross formation on the internal surfaces was noticed at the connection point between the cell and the cut-off, as shown in Fig. 2. This phenomenon is likely attributed to inadequate heat exchange during the printing process at that height of the structure.

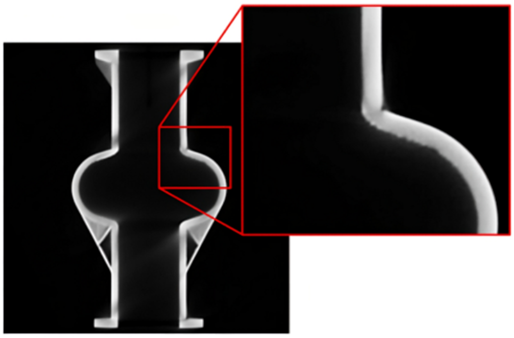

Figure 2: X-Ray tomograph of Nb cavity with detail on the iris section.

The prototypes of both Nb and Cu cavities successfully passed leak tests, demonstrating no signs of leakage in any of the produced samples. Resonance frequency testing was performed under as-built conditions and at room temperature. The measurements demonstrated excellent reproducibility of component performance through the LPBF process, with frequency values closely approaching the target frequency of 6 GHz. Small cavity measured 5.999 GHz, “big cavity 1” measured 5.995 GHz and “big cavity 2” 5.999 GHz.

Small AM samples were also made to assess its superconductive properties and yielded a critical temperature (T_C) of 9.15 ± 0.01 K. The samples also exhibited a Residual Resistance Ratio (RRR) value of 8. These findings align with the results obtained from conventionally processed niobium, indicating that the additive manufacturing process did not significantly impact the superconducting properties of the material [4].

Niobium cavities treatments

Two out of the three niobium cavities that were printed have been treated so far, specifically the "small cavity" and "big cavity 1." Additional treatments for the "big cavity 2" will be carried out in the near future.

The "small cavity" underwent a total of three vibro-tumbling treatments, with a combined duration of 390 minutes. After the VT surface treatments, the internal surfaces of the prototype exhibited significant smoothness, although some pitting-like defects remained visible. However, no new cracks or leaks were detected following the VT process. Surface roughness measurements were not taken due to the inability to access the cavity with a probe without cutting the prototype.

The "big cavity" underwent three vibro-tumbling (VT) processes and three electrochemical polishing (EP) processes. The VT processes lasted for a total of 480 minutes for the first two processes. As there were still surface defects present, the final VT process was extended for an additional 24 hours. The result was a smooth surface without any defects. Approximately 0.8 g of material was removed in total. After each VT process, a leak test was conducted, and all tests were successfully passed. No cracks were observed on the surface. Subsequently, a total of three EP processes were conducted using a solution of HF (46%): H_2SO_4 (98%) in a volume ratio of 1:9, resulting in the removal of 14g of material, equivalent to approximately 250 µm. The outcome was a surface with some signs of various-sized pitting, but overall, reflective with a notable macro e micro scale smoothing. Though, the down-skin effect was still present (Fig. 3). The leak detection test was successfully passed.

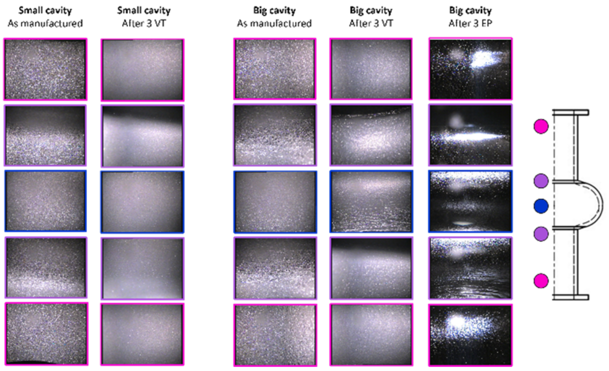

Figure 3: Internal inspections before and after treatments of Nb cavities.

Copper cavities treatments

A total of two copper cavities were produced using additive manufacturing (AM) (see Fig. 4). Both cavities underwent mechanical treatments at Rösler before being treated at INFN – LNL.

Figure 4: Cu printed T1 and T2 cavities.

The T1 cavity, after Rösler treatments underwent two electrochemical (EP) treatments prior to vibro-tumbling (VT) treatments. During the EP treatment, a solution of H_3PO_4 : Butanol (3:2 v.r.) was vertically flowed through the cavity. For the second treatment, the flow was reversed to improve the surface finish, as normally done with cavities at LNL. Vacuum leak checks were performed between treatments, and no leaks were detected. The EP treatments removed a total of 210 µm of material in the two processes lasting a total of 147 minutes. However, it was found that EP alone was not sufficient to smooth macro roughness of the internal surface, so the vibro-tumbling process was initiated. The VT process consisted of two runs and lasted a total of 95 minutes, removing an additional 39 µm of average material thickness. However, during the second vibro-tumbling process, the cavity broke at the iris point.

The CT scans revealed a vulnerable area in the internal fillet (iris) of the down-skin region, as shown in Fig. 5. This defect was observed in both the T1 and T2 cavities. It is likely that the defect resulted from the printing process, specifically, the layer thickness being too high, or the printing parameters not being adequately optimized for the down-skin region of these prototypes. This is particularly

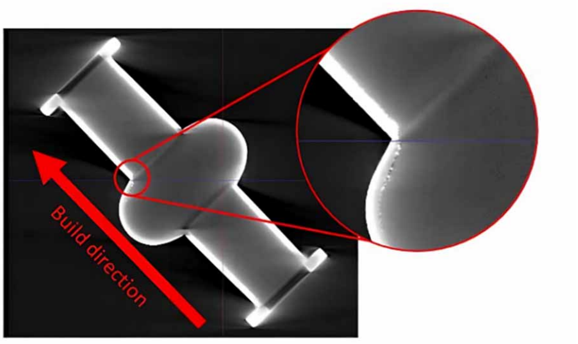

Figure 5: CT scan of cavity T1.

critical for the upper iris, as it lacks any supporting structure. However, this problem can be easily addressed by adding reinforcing structures in this area and optimizing printing parameters (as it was done with Nb cavities).

On the T2 cavity, the process was reversed, starting with mechanical polishing followed by electrochemical treatment. The mechanical polishing was performed by Rösler, resulting in a relatively smooth surface with some defects. Subsequently, one 60-minute EP treatments were carried out. A total of 94 µm of material was removed with EP. However, a leak was identified at the iris of the cavity. It is unclear whether this leak was caused by the mechanical or electrochemical treatment.

Visual inspections revealed that the combination of the two processes is highly effective in having better uniformity across the inner surface of the cavity (Fig. 6) [5].

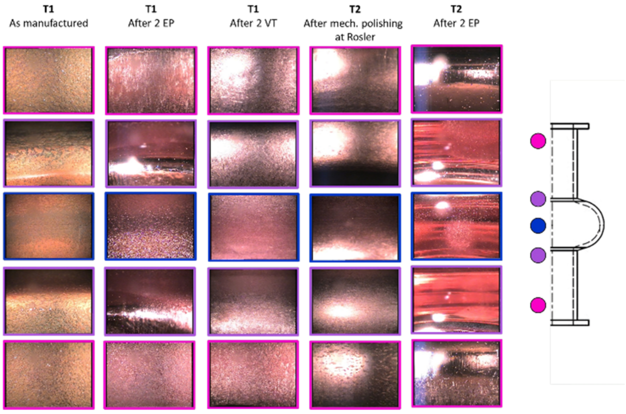

Figure 6: internal inspection of the copper cavities before and after each treatment.

CONCLUSIONS

A comprehensive fine-tuning campaign was conducted to optimize the density and surface finish of the printed objects, with a specific emphasis on the down skin parameters. The successful production of seamless pure niobium and copper 6 GHz accelerating cavities using LPBF additive manufacturing was achieved. Performance tests were conducted to assess the effectiveness of this manufacturing method, and highly promising results were obtained. Further surface treatments can be implemented to minimize surface roughness and address surface macro and micro defects, for e.g., Plasma Electrolytic Polishing, that might be able to effectively smooth internal surface of printed 6 GHz cavity [6, 7]. The research is ongoing, indicating a continued effort to advance the understanding and capabilities of additive manufacturing for accelerating cavities.

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