

NIOBIUM ALLOY C-103 FOR HIGH-PERFORMANCE SPACE APPLICATIONS – FIRST RESULTS WITH USING COLD SPRAY ADDITIVE MANUFACTURING – CSAM

Markus Brotsack⁽¹⁾, Ján Kondàs⁽¹⁾, Dr. Reeti Singh⁽¹⁾, Philippe Verlet⁽²⁾

⁽¹⁾ Impact Innovations GmbH, Bürgermeister-Steinberger-Ring 1, 84431 Haun/Rattenkirchen, Germany, Email: mb@impact-innovations.com

⁽²⁾ VLM Robotics, LASERIS Parc Scientifique et Technologique, 3 avenue Mayne Rabbit, Bâtiment n°3 CHERGUI, F-33 114 LE BARP, France

KEYWORDS: cold spray, additive manufacturing, CSAM, niobium, C-103

ABSTRACT:

Space propulsion applications require lightweight materials that can withstand high stresses at elevated temperatures. Niobium has a very low density compared to other refractory metals but high strength, i.e. a high strength-to-weight ratio. The material also shows high thermal conductivity and a low ductile-to-brittle transition temperature. This low transition temperature is advantageous for space applications because it shows excellent resistance to high-frequency vibrations at cryogenic temperatures. Furthermore, C-103, a niobium, hafnium, and titanium alloy, used for space propulsion applications, have a high melting point at around 2.350°C and show strong stability at elevated temperatures, too.

Currently, many investigations around C-103 material for in-space propulsion systems for satellites are happening in the space industry. This work shows the feasibility of Cold Spray Additive Manufacturing (CSAM) technology to produce a C-103 test part as a first step for future nozzle designs for different space applications. It is essential for the application to have as thin walls as possible to get down the take-off weight of the satellites. A test tube of around 125 mm in length, around 140 mm in diameter and 2 mm in wall thickness has been manufactured using CSAM. The manufacturing process will be presented in detail, in pictures.

It has been proven that C-103 can be sprayed with high deposition efficiency using cold spray technology. Deposition efficiency of around 92% with a 4 kg/h deposition rate was observed. Both properties make cold spray cost-effective for manufacturing in-space propulsion nozzles with C-103 niobium alloy. Furthermore, microstructural and mechanical properties of cold-sprayed C-103 will be investigated in near future.

1. INTRODUCTION: COLD SPRAY A SHORT OVERVIEW

Cold spray belongs to the family of thermal spray technologies (see Figure. 1). But there are a few major differences, which differentiates cold spray. This relatively new coating technology does not employ any flame, laser, burning or any other melting process. Cold spray uses metal powders, and the technology assures to accelerate individual metal powder particles up to three times supersonic speed. By reaching very high kinetic energies the metal particles are “hammered” onto a substrate. Applying this layer by layer, also near net shaped three dimensional parts can be generated. During the entire process the metal particles never reach the individual melting temperature, i.e. the metal particles stay “cold”. This property also assures that the substrate stays at moderate temperatures, i.e. any thermal effects, like deforming are minor compared to other thermal spray processes.

Due to the fact, that during the cold spray process no melting or burning is going along, the oxide content of the metal powder will not be changed during the manufacturing process, i.e. the coating on the substrate does have the same oxide content as the metal powder employed. This nature of the technology is an outstanding characteristic for applying corrosion resistant coatings.

Further characteristics of the cold spray process are:

- High deposition rates – up to 12 kg per hour
- High deposition efficiencies – up to 99% and above
- High dense coatings, i.e. low porosities – below 0,5%
- High thermal and electrical conductivities
- High hardness and homogeneity of coatings
- Multi-material stacking, including nonweldable materials
- Manufacturing of large-scale components

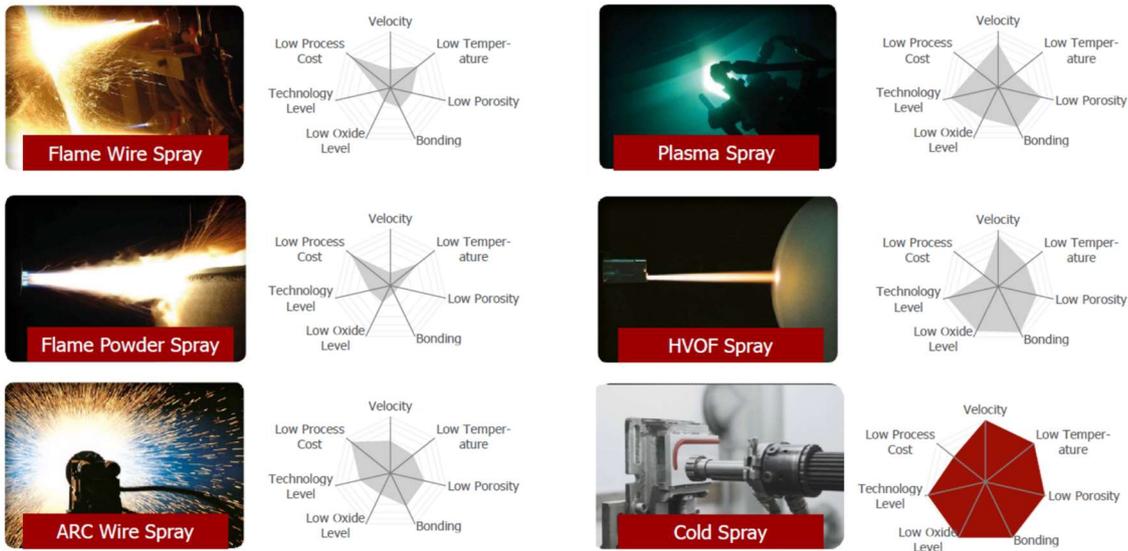


Figure 1: Cold spray another technology in the family of thermal spray processes [4].

2. COLD SPARY OF C-103 NIOBIUM ALLOY

This chapter investigates the world-wide first cold sprayed C-103 test tube. C-103 is a niobium alloy with some content of hafnium and titanium and some others in very low content. Cold spray properties have been determined and will be discussed. Finally, a large CSAM rocket nozzle will be introduced to proof the capability of cold spray being used for real shaped and sized space propulsions parts.

2.1. First C-103 cold sprayed test tubes

Finding out the behaviour of C-103 material while using it for cold spray additive manufacturing was the first goal while spraying on standard test tubes (see Figure 2). The niobium alloy has been applied on Al tubes to find out basic properties, like deposition efficiency (DE) and deposition rate (DR).



Figure 2: First test tubes to find out basic cold spray process parameters and deposition behaviour [13].

With this first set-up following main process parameters have been optimized:

- Temperature
- Pressure
- Feed rate

Also, three different C-103 powders from two different suppliers with different particle size distribution (PSD) were investigated. This is visible in Figure 2, please note the different colours and surface qualities.

The deposition efficiency after optimisation was very high, i.e. 92,4% DE was achieved. A powder feed rate of 4,83 kg per hour (i.e. 80,5 g/min) again proved that cold spray is an additive manufacturing (AM) technology with outstanding high material application rates. Taking into account the DE, a deposition rate (DR) of 4,46 kg/h (i.e. 74,4 g/h) could be realised. This is comparable with some other DED (Direct Energy Deposition) methods and is much higher compared to LPBF (Laser Powder Bed Fusion) systems.

2.2. Large sized simple test tube

The next step was to manufacture a more typical sized test tube being able to figure out what has to be adapted while scaling up the process. At stand alone tube with following geometry has been cold sprayed:

- Diameter: ~ 140 mm
- Length: ~ 125 mm
- Wall thickness: ~ 2 mm

At one end a flange was added with the following sizes:

- Length: ~ 4mm
- Thickness: ~ 8 mm

Following figure (Figure 3) shows the cold sprayed tube still with the inner mandrel, which has been removed afterwards. After the removal of the mandrel and some post machining the part looks like shown in Figure 4.



Figure 3: Large sized simple test tube on the mandrel [13].

From this part some tensile specimens were cut out, in two different states. Some specimens were machined from the as sprayed part, i.e. without any post heat treatment after cold spraying. A large cut out has been heat treated and from this part tensile specimens were cut to get mechanical properties after post heat treatment.



Figure 4: After mandrel removal and after post machining [13].

Finally, we got in total ten specimens, five without heat treatment and five with heat treatment (Figures 5 and 6).

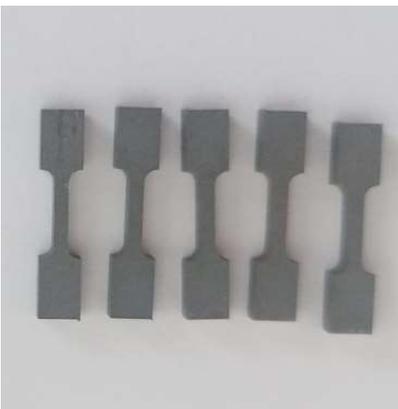


Figure 5: Five tensile specimens without heat treatment [13].



Figure 6: Five tensile specimens with heat treatment [13].

Just for reference following figure (Figure 7) shows the geometry of the specimens used.

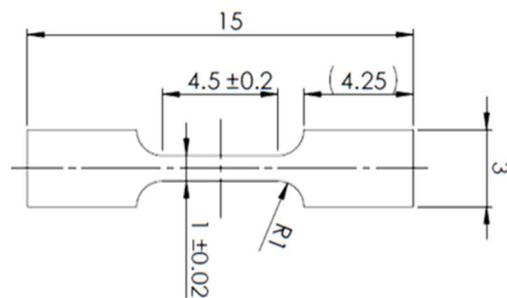


Figure 7: Geometry of cut out tensile specimens [13].

Unfortunately, the results are not available yet but are under investigation right now. The discussion of the results will be part of the continuation of this work.

2.3. CSAM of C-103 rocket nozzle extension

The final goal in this project was to additively manufacture a real sized rocket nozzle extension completely cold sprayed the niobium alloy C-103. The challenge in general is to generate large structures, i.e. large diameters and lengths, while keeping the wall thickness of the nozzle as thin as possible, to save as much weight as possible.

The dimensions, which finally have been realised with cold spray are:

- Max. Diameter: ~500 mm
- Length: ~600 mm
- Wall thickness: ~1,5 mm

By the way, with cold spray it is also pretty straight forward to adjust different wall thickness along the length of a nozzle. The thickness for example can vary within the nozzle between 1,5 mm to a few Millimetres.

With cold spray it is necessary to apply the material on a mandrel. There are first investigations using cold spray like other DED technologies to start the build up on a flat plate, but these trials are in very early stage.

Together with VLM Robotics in France a project could be realised to set-up the whole manufacturing process in one automated manufacturing line. Wire arc additive manufacturing was used to build up the mandrel made out of aluminium. In the same set-up and turning mechanism, a machining step cleaned and sized the mandrel according to the needed geometry. Finally, cold spray was used to generate the C-103 nozzle on the mandrel. The mandrel was chemical dissolved after finishing the cold spray process. A schematic process description can be found in the following figure (Figure 8).

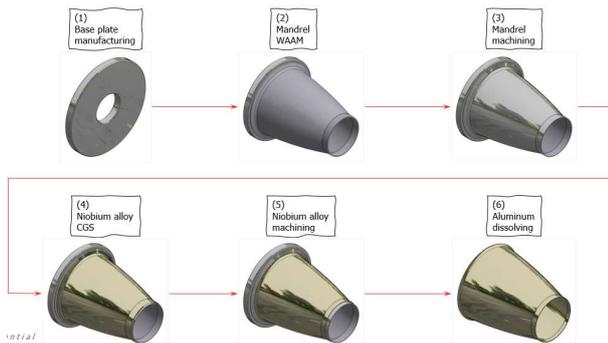


Figure 8: Schematic process flow of manufacturing a C-103 nozzle extension while using WAAM, machining, cold spray and mandrel removal [13].

The following shows the above shown schematic process with a few real images. Figure 9 illustrates the Al mandrel mounted on a horizontal turning lathe at the cold spray manufacturing cell.



Figure 9: Finished cold sprayed C-103 nozzle on Al mandrel [13].

Finally, the Al mandrel has been removed by chemical dissolution, see Figure 10. With this it is proven, that cold spray can be used to manufacture large rotational symmetric parts with minimised wall thickness and a buy to fly ratio close to one, which makes the process highly economic.



Figure 10: Final C-103 nozzle with Al mandrel removed, by chemical desolution [13].

3. SUMMARY, CONCLUSIONS AND OUTLOOK

For certain applications, especially rotational symmetric parts, like they are often used in space industry, cold spray is concerning feedstock, deposition efficiency and deposition rate a highly economic and fast additive manufacturing method. Chapter 1 is a short introduction to cold spray technology.

Chapter 2 of this investigation shows and discusses first results on cold spray properties, like deposition efficiency and deposition rate, of CSAM C-103 rotational symmetric test parts. This work impressively illustrates that cold spray in combination with other technologies can be automated to manufacturing space propulsion parts. This is getting more and more important, while looking at mankind's plans of the utilisation of low Earth orbit (LEO) and medium Earth orbit (MEO). The number of satellites will increase exponentially within the next few years, which implies that the number of rockets starts will increase accordingly, mainly for low and medium pay-load launchers. Not to forget the latest plans of different nations in the world to re-enter Moon surface and even beyond that to get a first step of a human being onto Mars' surface.

This work will continue, and right now mechanical properties are under investigation, like yield strength, tensile strength and adhesion strength of cold sprayed C-103. While determining these properties also a close look into the microstructure of the as sprayed material and after heat treatment will follow. Results will be available and reported in near future.

For interested readers some more CSAM examples of parts used for space propulsion can be found in chapter 5 at the end of this paper.

4. ACKNOWLEDGMENTS

Many thanks for the support of Philippe Verlet from VLM Robotics, which made sure to manufacture the aluminium mandrel while using WAAM technology. Last but not least the main contribution to this project was delivered by Ján Kondás from Impact Innovations, without his focused approach to the project this work wouldn't be at the stage as we have presented here.

This work has been financed by VLM Robotics and Impact Innovations GmbH.

5. FURTHER EXAMPLES OF CSAM PARTS FOR SPACE APPLICATIONS

This chapter just shows a few space propulsion components manufactured with the use of high-pressure cold spray technology. It's not intended to discuss detailed properties or sharing detailed manufacturing information. The intension is just to visualize the parts we do have in mind while talking about large space propulsion components.

5.1. Rocket combustion chamber

The combustion chamber is the core component of a rocket engine. For the inner liner material often CuCrZr is used.



Figure 11: Combustion Chamber component with helix-shaped cooling channels [4].

Figure 11 shows the demonstrator part manufactured with cold spray technology by Impact Innovations. Maximum diameter of the part is 240 mm, and the length is 295 mm. The chamber has been manufactured with helix-shaped cooling channels. The weight of this part after cold spraying was 36 kg and after machining around 32 kg.

5.2. Rocket nozzle

The challenge while producing this part was the stacking of two different materials. In this case the inner liner was made out of Cu and the outer jacket made out of Ni (see Figure 12).

The weight of this part is 41 kg as sprayed and after machining the part has 36 kg. The maximum diameter is 290 mm, and the length of the nozzle is

530 mm. The Ni flange thickness at the end with the smaller diameter is 35 mm.



Figure 12: Rocket nozzle after machining – manufactured by Impact Innovations [4].

5.3. Combustion Chamber - Manifold

For a proof of concept study the manifold part of a combustion chamber has been manufactured using high-pressure cold spray technology, i.e. EvoCSII from Impact Innovations. The project was performed together with the UK-based company airborne engineering. The design has been provided by airborne engineering and the manufacturing was performed by Impact Innovations. The final machined part is shown in Figure 13.

The maximum diameter is around 290 mm and the length, i.e. the thickness of the manifold is around 105 mm. Even the tube with flange, made out of In625, has been cold sprayed within the project. The length of the welded tube with flange is about 100 mm.



Figure 13: Combustion chamber manifold [4].



Figure 14: Inner structure of manifold [4].

Figure 14 shows the cut into parts manifold and gives some insight in the cooling channel structure, which has been realised. The figure also shows nicely the good bonding between the CuCrZr inner liner material with the In625 outer jacket. Both materials have been applied with one cold spray system, by only switching from one powder feeder filled with CuCrZr to a second feeder filled with In625 material.

5.4. Other examples from literature

This chapter just gives an overview of large scale manufactured cold spray parts, by well-known space companies.

ArianeGroup is evaluating cold spray technology for the combustion engines for the rocket Ariane 6. Due to the bimetallic capabilities of cold spray systems the inner liner (Cu-alloy) and the outer jacket (In625) have been cold sprayed (see Figure 15 and Figure 16 and [9]).



Figure 15: Cold spray of Cu-alloy – inner liner by ArianeGroup [9].



Figure 16: Cold sprayed In625 jacket on-top of Cu-alloy inner liner by ArianeGroup [9].

The sizes are in the range of ~700 mm height and ~400 mm in diameter. Just to get an idea about the dimensions.

NASA is using and testing cold sprayed structures a lot. One project was the Alpaca-Lunar lander. In this project the combustion chamber was successfully fire tested on the ground in April 2021. See Figure 17, which shows the cold sprayed engine before fire testing (see [10]).

More information can be found here:

[NASA 3D-Printed Engine Hardware Passes Cold Spray, Hot Fire Tests | NASA](#)



Figure 17: NASA – RDT Advanced Lander Propulsion Additive Cold Spray Assembly (ALPACA) [10].

Figure 18 shows the process of cold spraying of a coating with a Ni-based alloy onto a Cu-alloy inner liner, manufactured with a different additive manufacturing method.

The high kinetic energy applied to the metal particles assures a very good bonding to the Cu-alloy inner liner without melting, i.e. only using plastic deformation of the metal particles.



Figure 18: Cold spray bimetallic chamber structural jacket with Ni-based alloy [12].

6. REFERENCES

1. Tobias Schmidt, Kaltgasspritzen – Eine Analyse des Materialverhaltens beim Partikelaufrall und die daraus abgeleitete Prozessoptimierung, SHAKER Verlag, Aachen (2007)
2. P. Richter, L. Holzgaßner, J. Kondás, R. Singh, Conference Proceedings 11th Kolloquium Hochgeschwindigkeits-Flammspritzen/HVOF Spraying 25. Und 26. Oktober 2018, Gemeinschaft Thermisches Spritzen, Gilching, (2018)
3. W. Tillmann, J. Zajaczkowski, I. Baumann, M. Kipp, D. Biermann, Qualification of the low-pressure Cold Gas Spraying for the Additive Manufacturing of Copper-Nickel Diamond Grinding Wheels, Journal of Thermal Spray Technology, (2021)
4. Impact Innovations GmbH, internal presentations, (2021, 2023)

5. Julio Villafuerte (Ed.), *Modern Cold Spray, Materials, Process, and Applications*, Springer International Publishing Switzerland, Cham (2015)
6. Victor K. Champagne (Ed.), *The cold spray materials deposition process, Fundamentals and applications*, Woodhead Publishing Limited and CRC Press LLC, Cambridge and Boca Raton (FL), (2007)
7. C.M. Kay (ED), J. Karhikeyan (Ed.), *High Pressure Cold Spray, Principles and Applications*, ASM International, Materials Park (OH), (2016)
8. Pasquale Cavaliere (Ed.), *Cold-Spray Coatings, Recent Trends and Future Perspectives*, Springer International Publishing, Cham (2018)
9. Albus, J., Coipeau-Maia, V., Quadt, M., Beyer, S., Prampolini, M., Gagemann, G., Pichon, T. (2022). Ariane 6 Architecture and Future Evolution from a Materials and Structures Point of View. *73rd International Astronomical Congress (IAC), Paris, France, 18-22 September 2022*, IAC-22-C2.1.3
10. Gradl, P. (3rd March 2022). Metal Additive Manufacturing for Spaceflight. Presentation at: *Society of Manufacturing Engineers (SME) / Detroit and Houston Chapters*
11. Ajdelsztajn, L., Schoonover, J., Moran, E. (21st June 2023). Additive Cold Sprayed Ni Superalloys Low Cycle Fatigue, Presentation at: *CSAT / LSAAT 2023, Worcester, MA*
12. Gradl, P., Teasley, T., Protz, C., Garcia, M., Kantzos, C., Ellis, D. (2021). Advancing GRCop-based Bimetallic Additive Manufacturing to Optimize Component Design and Applications for Liquid Rocket Engines. *AIAA Propulsion and Energy Forum August 9-11, 2021*.
13. Impact Innovations GmbH, internal project data and presentation, October 2024 and ongoing update versions.