Characterization and Performance Evaluation of a Helium Recovery System Designed for Cold Spraying – Short form

Jean-Gabriel Legoux(1), Eric Irissou(1), Sylvain Desaulniers(2), James Bobyn(3), Bernard Harvey(1), Wilson Wong(3), Eric Gagnon(2) and Stephen Yue(3)

1 Industrial Materials Institute, National Research Council Canada, Boucherville, QC, Canada
2 Polycontrols Inc., 3650-A1, boul. Matte, Brossard, QC, Canada
3 Dept. of Mining and Materials Engineering, McGill University, Montreal, QC, Canada

Abstract: This paper describes and evaluates the performance of a Helium Recovery System (HRS) designed for cold spraying. A flexible, automated, full scale Helium Recovery System has been designed and installed in the McGill Aerospace Materials & Alloy Development Center Cold Spray Facility, located at and in collaboration with the National Research Council of Canada. The fully automated Helium Recovery System has been designed to recover helium from the cold spray chamber with sufficient purity (>99%) and flow capacity (5 to 220 Nm^3/h), allowing for a cost-effective operation by insuring a recovery rate of above 85%. In addition, a comparison of titanium coating properties obtained by using both He and N_2 as propellant gas is presented.

1 Introduction

Cold spray is a solid-state coating process in which powder particles are accelerated to high speed by the drag force of a supersonic gas stream and directed toward a substrate where they plastically deform and adhere to form a coating [1-3]. To enable bonding, the particles must reach a material-dependant critical velocity. For a wide diversity of materials, high deposition efficiency and high quality coatings are obtained using pre-heated air or nitrogen as the propellant. Gas pre-heating provides higher velocity to the spray particles and for most materials, increasing particle velocity leads to better coating microstructure and bonding [4-9]. However, increasing the propelling gas temperature also raises the risk of oxidation and/or nitridation due to higher heat input that it generates at the substrate surface [10, 11] which in turn may be detrimental for the design functionality of applied coatings. Helium provides therefore a solution to these issues since it is inert and allows reaching the higher particle velocity at a lower pre-heating temperature. Helium is however very costly compared to nitrogen, making it economically unviable for most applications unless recycled. A few papers and patents discuss and describe economic aspects as well as design principles of He recovery systems for cold spraying, but no comprehensive results were presented on actual efficiency and capability measured on an operating system [12-14]. A short description of an actual operating pressure swing adsorption (PSA-) based HRS is given in ref [7]. They report a recovery rate of higher than 70% while in a more recent paper the same author group reports 85% recovery [15].

In this paper we present initial results on the operating performance and efficiency of a HRS designed to investigate the industrial potential of the cold spray process using helium as the propelling gas. Polycontrols Technologies Inc. was mandated to design and integrate a Helium Recovery System to the cold spray laboratory of the McGill Aerospace Materials & Alloy Development Center (MAMA-DC) located within the Industrial Materials Institute of the National Research Council of Canada (NRC-IMI). The Helium Recovery System was specified to be suitable to recover filtered helium from their two air tight cold spray chambers with flow rates from 5 to 220 Nm^3/h and to provide recycled helium at 40 bar with a purity of at least 99% for runs as short as 2 minutes and as long as 7 hours.

2 Experimental

Spray booth design

The spray operation is conducted into a 28 m^3 enclosure equipped with robots to enable either gun or specimen movement. A small 3 m^3 rectangular spray hood is installed inside the spray room. The process parameters are controlled by commercial cold spray equipment and the operation of the helium recovery system does not require any input or modification to the spray equipment. Even though the system is using standard thermal spray equipment some important differences are present. The first one is that the dust collecting system needs to be able to work in closed loop. Moreover the complete system needs to be gas tight since any leak would result in a loss of the precious Helium molecules. In order to achieve a high degree of gas tightness a medical grade dust collector system was selected. At start-up of the system, the spray enclosure, the inside of the dust collector and the ventilation ducts are filled with air. The air in this dead volume has to be extracted partially in order to be able to start recycling Helium.

Therefore, the start-up procedure consumes an important quantity of Helium that is directly proportional to the dead volume of the spray booth. In order to minimise this dead volume, the dust collector was located as close as possible to the spray area. Also the spray enclosure volume can be modulated in function of the needs. Since most of the development work is performed either while taking measurements on the particles jets or for producing small coupons, the spray hood can be sealed. The dead volume is then reduced by 25 m^3. In this configuration, because of the space limitation, the gun has to be stationary and coupons are translated in front of it using a compact robot.

Operating principles of the Helium Recovery System (HRS)

The Helium Recovery System is connected to the ventilation ducts after the gas stream is cleaned from particles. The Helium Recovery System consists of a gas separation unit, compressors, storage tanks and different analysers and instruments. The system is fully automated and is designed to adapt itself to the instantaneous spray conditions, namely gas flow and pressure. This enables the system to be transparent to the operator; that is no intervention is required from the operator who can concentrate on the spraying.

The gas separation system is designed to separate gas mixtures by means of the Pressure Swing Adsorption (PSA) process. This process takes advantage of the selective adsorption characteristics of different gases by zeolite adsorbents when subjected to varying pressures. The mixed gases which come from the cold spray projection chambers (Feed Gas) are made to flow upwards vertically through a tall column of adsorbent material at high pressure. The easily adsorbed components of the mixture (‘heavy’ components) are retained by the adsorbent while the ‘light’ components (the Helium in occurrence) are released from the top of the column as purified (Product) gas. Before the adsorbent material becomes saturated by the ‘heavy’ gas components, product delivery is halted and the pressure is released from the bottom of the adsorbent column (‘bed’). The lower pressure releases the heavy gases from the zeolite and they are forced to flow out of the system as waste (exhaust) gases. The adsorbent bed is then re-pressurized and is ready to repeat the cycle.

The inherently intermittent nature of the process means that a single adsorbent bed can only deliver purified product gas for a portion of its cycle. To ensure a continuous supply of product gas, the system is equipped with nine adsorbent beds which are cycled in a balanced, sequential manner through identical operating cycles. In addition to providing a continuous supply of product gas, the operating cycle also includes advanced features such as pressure equalization between beds to minimize energy losses and an advanced flow control system to allow adjustment for peak operating performance.

Two compressors assure continuous supply of gases through the entire system. The first one, located on the feed line, assures optimum pressure to the PSA and the second one, located on the product line, assures helium storage and supply to the cold spray gun. To meet the specific requirements of the MAMA-DC and NRC-IMI research programs, both compressors are equipped with variable speed controls and, receiver tanks capacities have been increased to aid separation performance and to minimize flow and pressure surges.

The Helium Recovery System is also equipped with two thermal conductivity gas analysers which monitor the helium concentration in the projection chamber, feed line, product line and exhaust line. These analysers, paired with optimisation algorithms, ensure a stable process even under changing conditions. They also allow the improvement of the recovery rate by controlling the enrichment of the atmosphere of the projection chamber. The sub-ambient (helium enriched) atmosphere created in the chamber is maintained at constant pressure slightly below atmospheric pressure. This delicate equilibrium allows loading and unloading of the samples without losing helium during the opening of the access port, and without having significant impact on the Helium Recovery System.

The Helium Recovery System is provided with instrumentation for process management as well as for automatic shut down and start-up duties. These devices are connected to a supervisory and control PLC. The supervisory and control system has all the necessary information to control the PSA in order to adjust the flow and the purity of the Helium as required by the cold spray system, and to assure the supervision of secondary systems such as compressors, thermal conductivity gas analysers, interface with the cold spray system, system enrichment and special atmosphere in the projection chamber. Supervision man-machine interface software provides clear and intuitive control of the Helium Recovery System, diagnostics, alarms, automatic report generation and easy integration into other control systems.

3 Results and discussion

3.1 Performance evaluation of the Helium Recovery System designed for cold spraying

During its commissioning tests for integration in MAMA-DC and NRC’s research projects for the cold spray process using helium, the Helium Recovery System has undergone several studies to quantify its performance under different operational scenarios. The following sections show the process performance, repeatability and consistency under different operating conditions. For all these descriptions the following nomenclature was adopted: The feed gas refers to the gas entering the Helium Recovery System; The feed Purity represents the helium concentration included in the gas mixture which enters the Helium Recovery System. The Product gas is the gas that is exiting the Helium Recovery System through the cold spray gun. Thus Product flow rate corresponds to the flow of recovered and recycled helium provided to the cold spray gun. The demand of gas is the gas flow that is actually fed to the cold spray gun.

The efficiency rate refers to the volume of recycled helium consumed divided by the total volume of helium used by the cold spray process. The global efficiency ratio includes all losses from the Helium Recovery System, dust collector, spray chambers, and loading/unloading of samples through the load-lock. For efficiency computation all data excludes the start-up and stabilization phases of the Helium Recovery System.

Start-up

Figure 1 shows the helium concentration evolution during the start-up of the recycling operation. It is worth noting that this is the phase of the process during which the system shows its lowest efficiency. This is caused by the fact that the purification operation needs to be performed with a gas stream containing at least 60% of helium. Thus during the initial step of the process even if the cold spray gun is fully operational, the helium is mainly filling up the spray booth and the dust collection system. It is worth mentioning that the optimization of the control loop of this phase leads to the virtual elimination of fluctuation in the product purity.

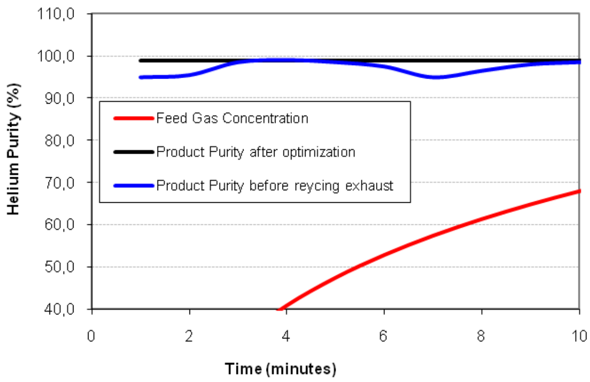

Figure 1: Evolution of helium concentrations at the start-up of the system.

Permanent regime

About 15 minutes after the initial start-up, the Helium Recovery System reaches a permanent regime. During this stage the helium losses are limited to the leak rate of the facility. Figure 2 shows the helium feed and product purity variation during this stage. It should be noted that because of the infiltration of some air inside the facility the feed gas concentration is gradually reduced. However this decrease in purity is relatively slow and gradual. The concentration (>99%) and production rate of the purified helium is not affected by this variation. During this stage the helium recovery efficiency is very high and can reach 95%.

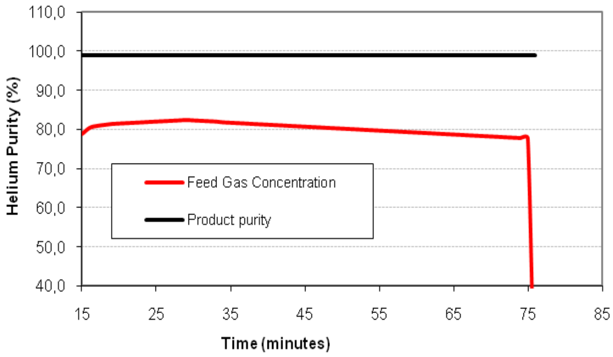

Figure 2: Evolution of the helium concentration in the feed gas and the product in permanent regime.

Variation of spray conditions

Figure 3 shows the variation of flow rates and gas purity when sudden changes in the gas flow rate are imposed by the operator. It should be noted that the purity of the product is not affected by flow variation and remains close to 100%. Note also that the maximum flow rate that can be reached by the current version of the Helium Recovery System is 150 Nm^3/h. When the spray system requires a higher flow rate, the system can provide it, but it is then digging into its reserve, and the amount of gas exceeding this limit is vented out leading to a reduced global efficiency. This limit is linked to the feed compressor capability and thus would be easily improved by the addition of an auxiliary compressor.

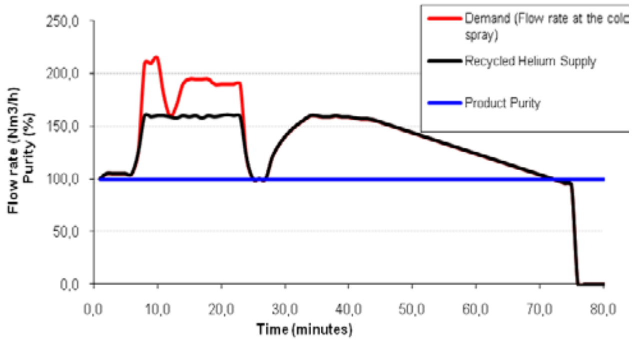

Figure 3: Recycled Helium Supply vs varying Demand (Variation of flow rate at the cold spray gun)

System efficiency

Table 1 shows the values of the feed gas purity, the product flow rate, the global efficiency including the start-up phase, and the efficiency rate in permanent (steady-state) regime, for the design basis and initial results, and after leak proofing improvement and system optimization.

The increase of the helium feed purity to values closer to the design basis is the result of mechanical changes made to the industrial dust collector. Since the collection collar of the Helium Recovery System is located on the return of the dust collector, which has a relatively large dead volume and a complex geometry, a significant portion of the light gas initially recovered from the projection chamber was released to the atmosphere instead of being returned to the Helium Recovery System. Improvements made to seals significantly increased the overall recovery rate of the system.

Improvements in the efficiency are also the results of an optimization of flow paths, implementation of control loops that adjust the cycle speed of the PSA according to the feed purity and controlling the enrichment of the atmosphere in the projection chamber.

Some tests have also been conducted with the addition of a vacuum pump to clean the beds of the PSA. However, analysis of the data collected has not shown conclusive results, therefore more tests will be needed before any modifications are made.

It is beyond the scope of this paper to conduct a precise cost comparison analysis. However assuming the helium cost is 10 times that of nitrogen, current results show that for continuous operation or for long batches, the cost of operating a cold spray system using helium instead of nitrogen can be very similar. If it is assumed that nitrogen and helium are used at a similar gas flow rate, an efficiency of helium recovery close to 90% leads to identical cost. Of course a precise analysis will need to be conducted taking into account the large difference in capital investment, deposition efficiency and perhaps more importantly the capability of producing coatings or coating quality that are not possible using nitrogen only.

Table 1 – Summary of runs performed with the Helium Recovery System (HRS)

	Feed Purity (%)	Product Flow Rate (Nm^3/h)	Global Efficiency	Perm. regime Efficiency
Design Basis	85	220	80%	85%
Initial Results	65-70	150-160	73%	80-85%
Optimized results	78-80	150-160	88%	90-95%

3.2 Coating evaluation using recycled helium as compared to nitrogen as propelling gas

In order to characterize the behavior of the cold spray system using helium, titanium powder was used as a model feedstock material and coatings were produced with both He and N_2 as propelling gas, for comparison. The cold spray system used was a Kinetiks 4000 (CGT GmbH, Germany). The process conditions were selected to obtain the highest particle velocity possible with each gas. For nitrogen, the MOC24 nozzle (CGT GmbH, Germany) was used and the propelling gas pressure and temperature were set to 40 bar and 800°C, respectively. For helium, the ASB VH70 nozzle (ASB Industries Inc., USA) was used and the propelling gas pressure and temperature were set to 40 bar and 350°C, respectively. Particle velocity was measured using a DPV2000 (Tecnar Automation Inc., Canada). The coatings were deposited onto mild steel plates using a similar spray pattern to that in ref. [16]. The plasma atomized Cp-Ti powder (Raymor, Canada) was used and a complete characterization of this powder is presented elsewhere [17].

Particles velocity measurement

The particle velocity distributions for the parameters selected in this study are shown in Figure 5.

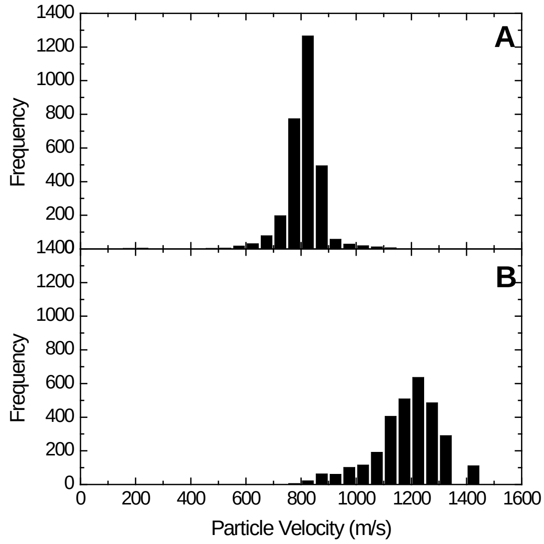

Figure 5: Particle velocity distribution using nitrogen (A) and helium (B)

The measured average velocity (standard deviation) were 805 (74) and 1173 (126) m/s for nitrogen and helium as propelling gas, respectively. The velocity value obtained with nitrogen is similar to that reported by Zahiri et al [18] (790 m/s). However, it is worth noting that the velocity obtained using helium is significantly higher than the reported 900 m/s in ref. [18], more likely due to the difference in nozzle configuration.

Coatings

Coatings were produced with the conditions used to monitor the particle velocity. For both conditions, deposition efficiency was measured and reached 100 %; however the microstructures of the Ti coating are quite different. These microstructures are shown in Figure 6. The coating produced using nitrogen remains porous (~2%) while the one produced using helium is fully dense. The differences between both coatings are described in more detail in ref. [16].

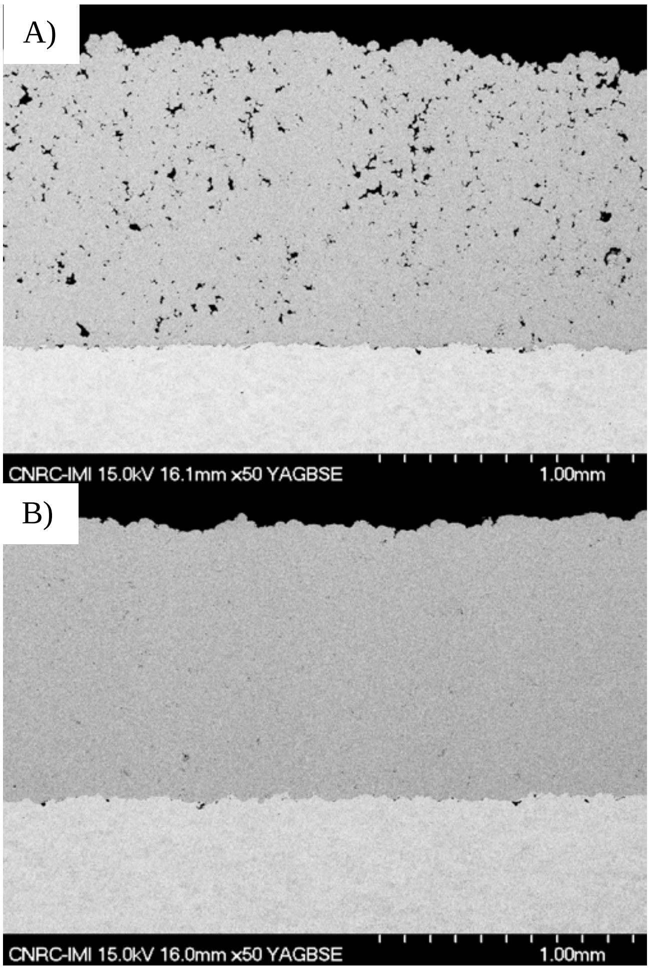

Figure 6: Micrograph of the Ti coatings made using nitrogen (A) or helium (B)

4 Conclusions

This paper describes and evaluates the performance of a Helium Recovery System (HRS) designed for cold spraying. It was demonstrated that:

- It is possible to integrate an efficient helium recovery system with a standard thermal spray configuration.
- It is possible to operate in a continuous mode an automated helium recovery system having an efficiency higher than 90%
- Using helium instead of nitrogen enables an increase in titanium particle velocity of 400m/s leading to improved coating quality.

Acknowledgements

The helium recovery system and the cold spray equipment were provided by CFI project number 8246, awarded to McGill University.

References

1. R.G. Maev, and V. Leshchinsky, Introduction to Low Pressure Gas Dynamic Spray: Physics & Technology Wiley-VCH Verlag GmbH 2008.
2. V.K. Champagne, The cold spray materials deposition process: Fundamentals and applications, Woodhead Publishing Limited, 2007.
3. A. Papyrin, V. Kosarev, K.V. Klinkov, A. Alkhimov, and V.M. Fomin, Cold Spray Technology, A. Papyrin. Ed., Elsever, 2006.
4. K. Balani, T. Laha, A. Agarwal, J. Karthikeyan, and N. Munroe, Effect of carrier gases on microstructural and electrochemical behavior of cold-sprayed 1100 aluminum coating, *Surface and Coatings Technology*, 2005, **195** (2-3), p. 272-279
5. H.J. Kim, C.H. Lee, and S.Y. Hwang, Fabrication of WC-Co coatings by cold spray deposition, *Surface & Coatings Technology*, 2005, **191** (2-3), p. 335-340
6. C.J. Li, and W.Y. Li, Deposition characteristics of titanium coating in cold spraying, *Surface & Coatings Technology*, 2003, **167**, p. 278-283
7. J. Pattison, S. Celotto, R. Morgan, M. Bray, and W. O'Neill, Cold gas dynamic manufacturing: A non-thermal approach to freeform fabrication, *International Journal of Machine Tools & Manufacture*, 2007, **47** (3-4), p. 627-634
8. S.H. Zahiri, C.I. Antonio, and M. Jahedi, Elimination of porosity in directly fabricated titanium via cold gas dynamic spraying, *Journal of Materials Processing Technology*, 2009, **209** (2), p. 922-929
9. S.H. Zahiri, W. Yang, and M. Jahedi, Characterization of cold spray titanium supersonic jet, *Journal of Thermal Spray Technology*, 2009, **18** (1), p. 110-117
10. E. Irissou, J.-G. Legoux, A.N. Ryabinin, and C. Moreau, How Cold is Cold Spray? An Experimental Study of the Heat Transfer to the Substrate in Cold Gas Dynamic Spraying., *Thermal Spray 2008: Proceedings of the International Thermal Spray Conference*, 2008.
11. S.Guliziaa, B.Tiganis, M.Z.Jahedi, N.Wright, T.Gengenbach, and C.MacRae, Effects of Cold Spray Process Gas

Temperature on CP Titanium Structure, *ITSC2009*, 2009, p. 237-242.
12. F. Lauricella, and S. Jaynes, Helium recovery: design considerations for cold spray systems, *ITSC 2003: International Thermal Spray Conference*, ASM International, 2003, p. 113-116.
13. S.E. Jaynes, System and process for gas recovery, Praxair Technology, I. N. C., 2006.
14. E. Irissou, J.G. Legoux, A.N. Ryabinin, B. Jodoin, and C. Moreau, Review on cold spray process and technology: Part I - Intellectual property, *Journal of Thermal Spray Technology*, 2008, **17** (4), p. 495-516
15. J. Pattison, S. Celotto, A. Khan, and W. O'Neill, Standoff distance and bow shock phenomena in the Cold Spray process, *Surface and Coatings Technology*, 2008, **202** (8), p. 1443-1454
16. W. Wong, E. Irissou, J.-G. Legoux, and S. Yue, Influence of helium and nitrogen gases on the properties of cold gas dynamic sprayed pure titanium coatings, *ITSC2010*, 2010.
17. W. Wong, A. Rezaeian, E. Irissou, J.-G. Legoux, and S. Yue, Cold Spray Characteristics of Commercially Pure Ti and Ti-6Al-4V, *Advanced Materials Research*, 2010, **89-91**, p. 639-644
18. S.H. Zahiri, W. Yang, and M. Jahedi, Characterization of Cold Spray Titanium Supersonic Jet, *Journal of Thermal Spray Technology*, 2008, p. 1-8