Fundamental Investigation into the Effects of In-process Heat Treatment in Cold Spray

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Abstract

Cold spray is a technology with great potential for additive manufacturing applications. Due to the high levels of plastic deformation experienced by the powder during the coating process, any deposit will require heat treatment post-spraying to improve ductility and fatigue strength. In extreme cases, the residual stresses from coating can cause delamination or compromise the bond strength when subsequent cold spray layers are deposited. This work details the use of a commercial CO_2 laser cutter to perform a surface heat treatment on single lines of cold sprayed aluminium, to relieve residual stresses. The effect of laser power and traverse speed on material hardness is quantified, and compared with as sprayed deposits. The results shown in this work demonstrate the potential for in-process heat treatment to reduce post-processing time and improve coating quality by reducing residual stresses.

Introduction

Cold spray is a process where powdered material is accelerated to high velocity using a supersonic nozzle, after which the material impacts upon a substrate and deposits primarily through plastic deformation, with no melting or other significant thermal changes required [1]. Due to the large amount of plastic formation required, work hardening will occur within the material, often causing residual stresses within the deposit [2]. The effect of residual stresses on coating adhesion has been recognized as an issue which must be understood to allow for the widespread application of cold spray [3].

The magnitude of the residual stresses within a cold sprayed deposit increase with increasing particle velocity, increasing particle size, and as the angle of deposition approaches 90° [4]. Some investigations have examined the effect of substrate preheating on residual stresses, finding that the substrate preheating does not appear to affect the level of residual stresses after deposition, but the deposition of subsequent layers seems to cause tensile residual stresses between the deposited layers [5], [6].

Both tensile and compressive residual stresses can be generated in cold sprayed coatings [7], and where compressive stresses are present they do not always deliver the anticipated improvements in fatigue strength that would be commonly expected [8]. Some attempts have been made to overcome the poor fatigue properties of cold spray by using pre and post process peening [9]. Post-spraying heat treatment can relieve residual stresses in a variety of cold sprayed materials [10]–[13], but these heat treatment processes require both time and infrastructure [14], and can only be carried out after the cold spray deposition is complete.

Lasers have been used in-process in cold spray to heat the deposition zone, increasing deposition efficiency and allowing for the deposition of more difficult materials [15]. There has been work into post-spraying laser heat treatment by Carlone et al., who studied the effect of laser heat treatment on titanium. It was observed that an oxide layer and heat affected zone formed after heat treatment using a 220 W diode laser with a 2 mm diameter spot size, giving a power density of approximately 70 W/mm^2 [16]. Astarita et al. carried out laser surface remelting of titanium using the same laser [17]. Carlone et al. found that there was an optimum amount of energy input for effective heat treatment, with lower heat input levels having no effect, while higher heat input levels causing cracking of the coating. The thermal mass of the substrate and coating was also shown to change the heat treatment results obtained, with samples with higher thermal mass showing less effects from heat treatment due to lower peak temperatures during treatment.

This paper will investigate the potential for using a CO_2 laser for heat treatment of a variety of cold sprayed materials. While this heat treatment will be carried out post process, it is intended to give insight into the possibility of implementing a laser system for in process heat treatment during spraying operations.

Experimental Setup

Material Selection

In order to assess the effect of surface heat treatment, a range of cold spray materials were chosen, with grades and powder suppliers as follows:

1. Aluminium 6061 – LPW technology (Runcorn, United Kingdom)
2. Copper (commercially pure) – Safina AS (Vestec, Czech Republic)
3. Stainless steel (grade 304) – LPW Technology
4. Titanium (grade 2) – Active Metals (Sheffield, United Kingdom)

Cold Spraying Operations

The four materials chosen were deposited onto aluminium substrates using the parameters shown in Table 1. The cold spraying was carried out in Trinity College Dublin using a bespoke cold spray machine (as detailed in [18]), with a traverse speed of 200 mm/s and nozzle standoff of 40 mm. Single tracks were deposited for analysis and heat treatment.

Table 1: Cold spray parameters

Material	Carrier gas	Gas pressure *barg*	Gas heating *°C*
Al 6061	He	20	100
Cu	He	30	550
SS	He	20	Room
Ti	He	20	Room

Laser Heat Treatment Operations

A BRM 90130 CO_2 laser cutter (BRM Lasers, Winterswijk, The Netherlands) was used to carry out heat treatment operations on the cold sprayed tracks. No focusing optics were used, with the unfocussed spot size of approximately 6 mm diameter directed in a zigzag pattern to cover the width of the cold sprayed track. A constant laser power of 95 W was used for all heat treatment operations, with a matte black paint applied to the samples prior to heat treatment to minimize reflectivity and thus maximize the amount of energy delivered by the laser beam. In this configuration the laser has a power density of approximately 3.36 W/mm^2. The heat treatment parameters used are shown in Table 2. An initial investigation into the effect of traverse speed was carried out on Al 6061, with the other materials being tested at a single traverse speed.

Table 2: Laser heat treatment parameters

<table>
<tr><th>Material</th><th>Traverse speed</th><th>Number of passes</th></tr>
<tr><td>Al 6061</td><td>2.5, 5, 10, 15 mm/s</td><td rowspan="4">1, 5, 10</td></tr>
<tr><td>Cu</td><td rowspan="3">10 mm/s</td></tr>
<tr><td>SS</td></tr>
<tr><td>Ti</td></tr>
</table>

Material Characterization

Heat treated samples were mounted and polished to allow for inspection and hardness testing. Vickers hardness with a 100 g load and 10 s dwell time was used, with measurements being made close to the surface of the deposit, close to the substrate, and at a position approximately halfway between the substrate and surface. Previous studies have shown that microhardness values decrease with heat treatment [19]. Any porosity or particle boundaries were avoided to ensure that the hardness measurement was a measure of the "bulk" hardness rather than interparticle boundaries. Past studies have shown that techniques such as nanohardness can have large variation in results due to the weaker interparticle boundaries being measured [12].

Results and Discussion

Aluminium 6061

The results of a preliminary investigation into the effect of traverse speed are shown in Figure 1. For the 2.5 mm/s traverse speed it was observed that more than one pass caused surface melting on the sample, so these were not analyzed further. As no shield gas was used, these samples would be expected to show high levels of oxidation and be unsuitable for practical applications. In order to avoid melting or recrystallisation during heat treatment, it was decided to use a traverse speed of 10 mm/s for the remaining analysis.

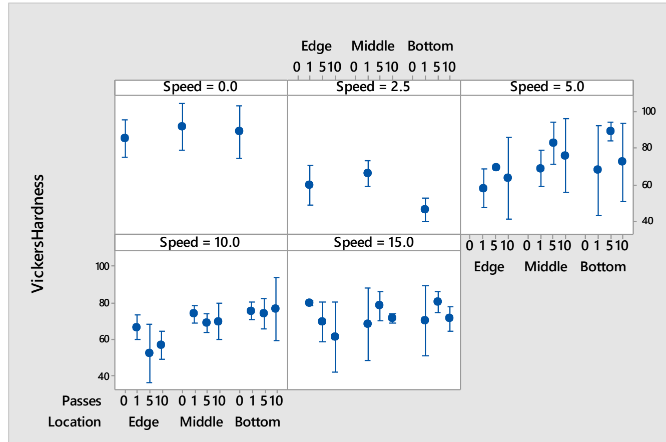


Figure 1: Coating hardness results for Aluminium 6061, for all traverse speeds. Error bars are 95% confidence intervals. Edge refers to the coating edge, while bottom refers to near substrate.

Figure 2 shows the results for aluminium 6061 for the 10 mm/s traverse speed only, with the zero passes data points being those of the as-sprayed samples. It is clear that at the coating edge there is a reduction in hardness due to laser heat treatment, with the effect reducing in magnitude in the middle of the deposit, and further reducing near the substrate. The lowest mean hardness values are achieved after 5 passes, after which there is a slight increase in the mean in all locations, possibly indicating that repeated heating/cooling cycles are having an effect beyond a simple annealing process.

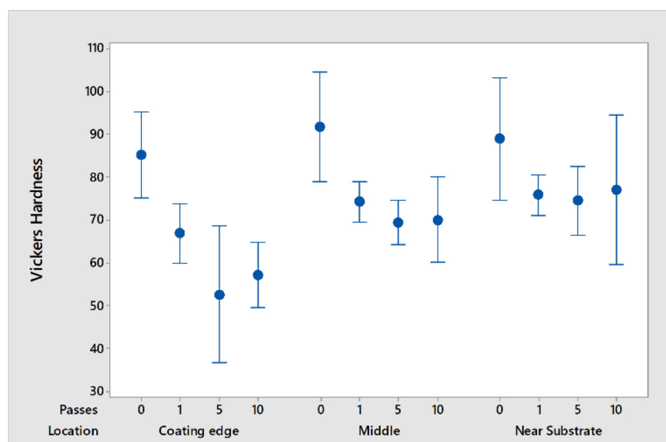
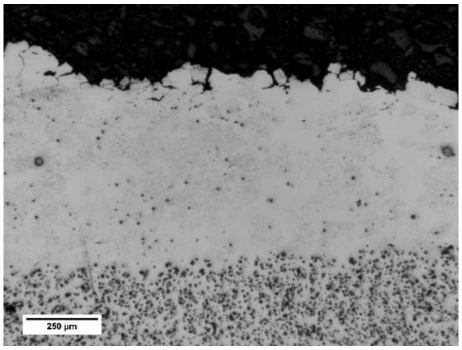
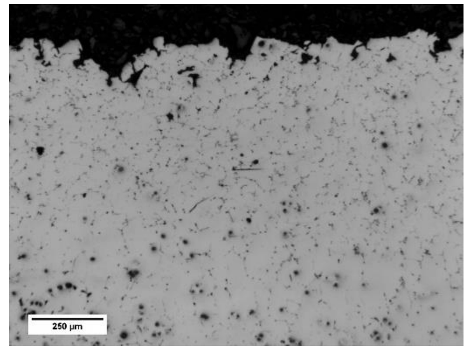


Figure 2: Coating hardness results for aluminium 6061 samples. Error bars are 95% confidence intervals.

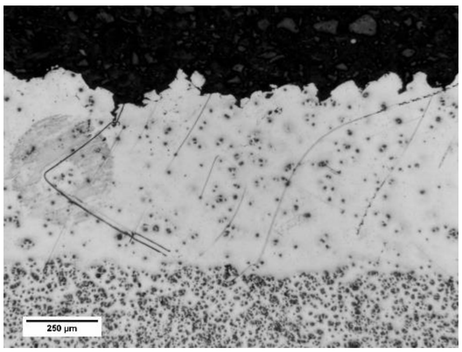
Figure 3 (overleaf) shows the polished cross sections of the aluminium 6061 samples for all traverse speeds. There does not appear to be any significant changes in material structure or porosity levels as a result of laser heat treatment.



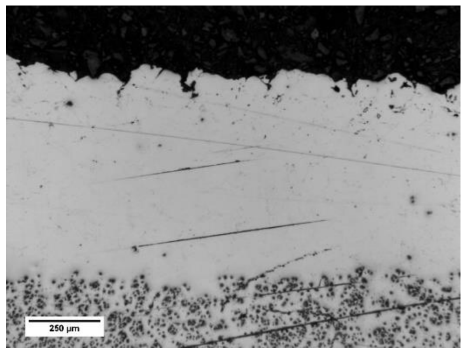
(a) As sprayed



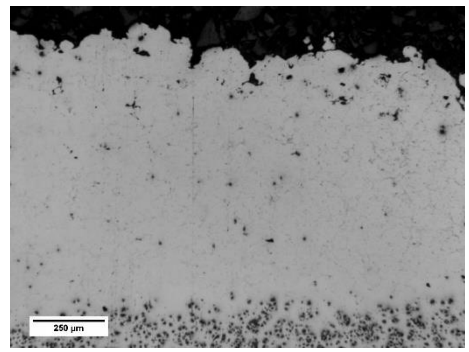
(b) Heat treated, 2.5 mm/s, 1 pass



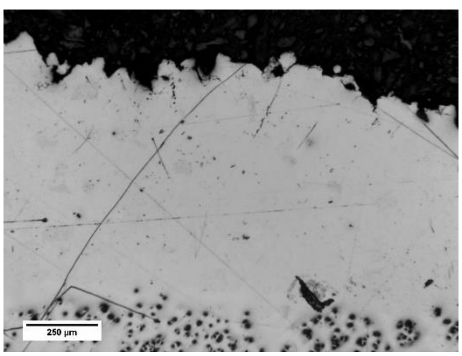
(c) Heat treated, 5 mm/s, 1 pass



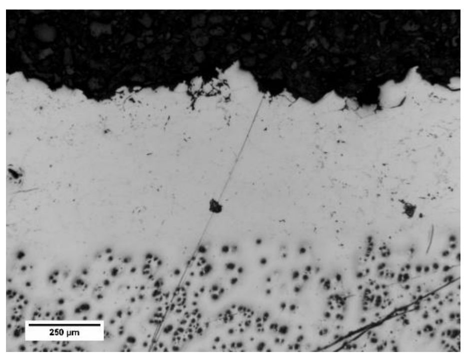
(d) Heat treated, 5 mm/s, 5 passes



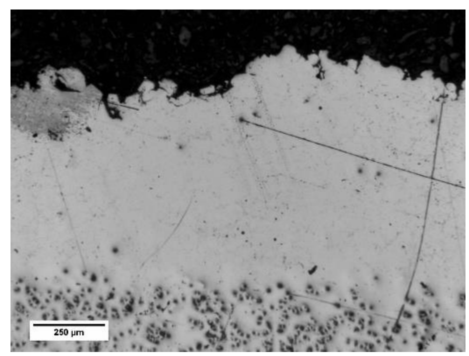
(e) Heat treated, 5 mm/s, 10 passes



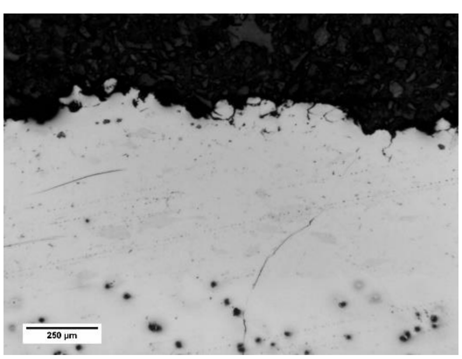
(f) Heat treated, 10 mm/s, 1 pass



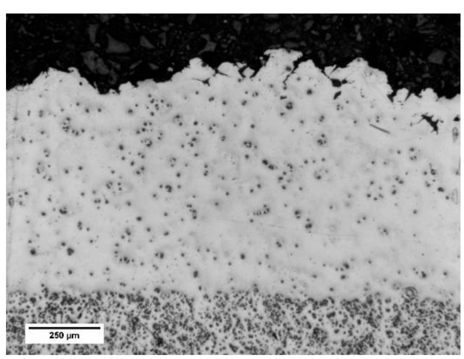
(g) Heat treated, 10 mm/s, 5 passes



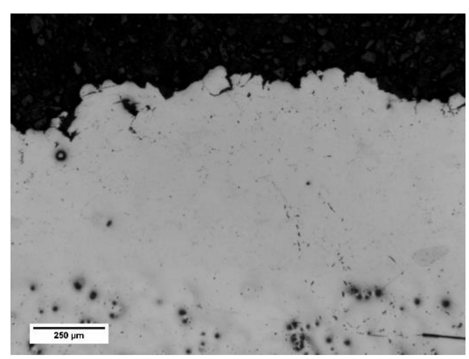
(h) Heat treated, 10 mm/s, 10 passes



(i) Heat treated, 15 mm/s, 1 pass



(j) Heat treated, 15 mm/s, 5 passes



(k) Heat treated, 15 mm/s, 10 passes

Figure 3: Cross sections of mounted and polished aluminium 6061 samples after laser heat treatment

Copper

Figure 4 shows the hardness results for copper samples for a laser traverse speed of 10 mm/s. Similar to the aluminium 6061 results, there is a reduction in hardness due to laser heat treatment, with the minimum mean hardness in all locations reached after one coating pass. This indicates a more complex heat treatment process at work than annealing alone.

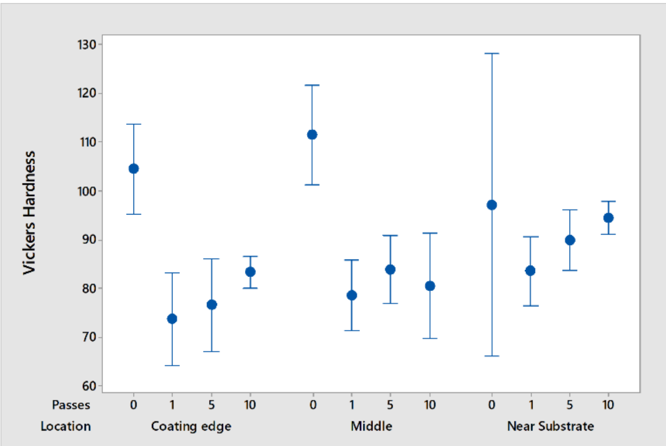
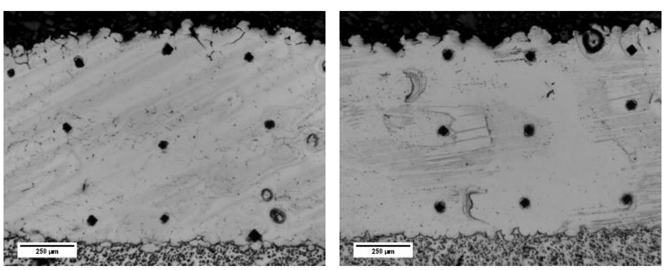
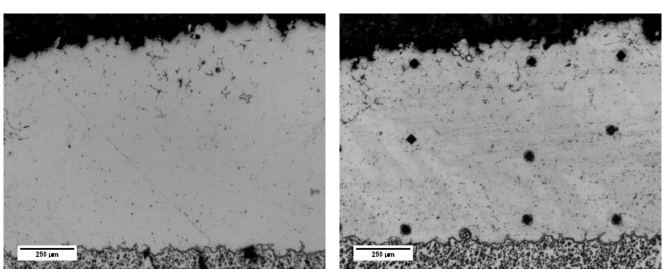


Figure 4: Coating hardness results for copper samples. Error bars are 95% confidence intervals.

Figure 5 shows polished cross sections of the copper samples. As for the aluminium 6061 samples, there does not appear to be any change in porosity level or material structure as a result of the heat treatment process.



(a) As sprayed (b) Heat treated, 1 pass



(c) Heat treated, 5 passes (d) Heat treated, 10 passes

Figure 5: Cross sections and mounted and polished copper samples after laser heat treatment

Stainless Steel

Figure 6 shows the hardness results for stainless steel samples for a laser traverse speed of 10 mm/s. Unlike the aluminium 6061 and copper samples, there is no clear effect as a result of heat treatment, for any location in the coatings.

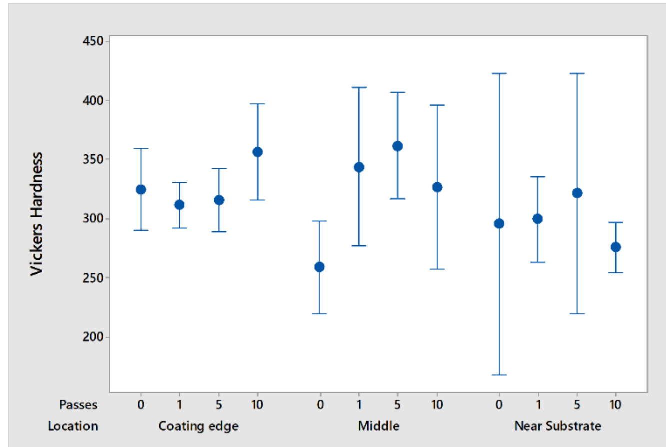
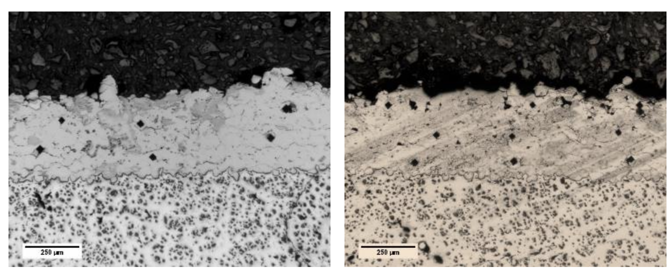
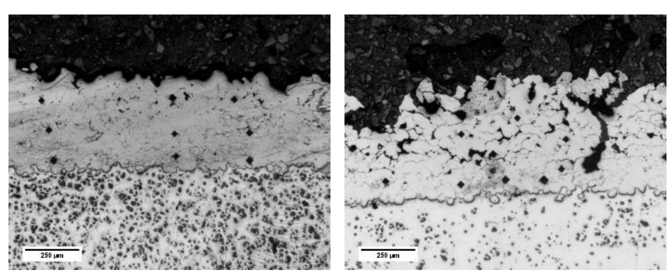


Figure 6: Coating hardness results for stainless steel samples. Error bars are 95% confidence intervals.

Figure 7 shows polished cross sections of the stainless steel samples. While the as sprayed and 1 and 5 pass samples do not show any change in material structure, the 10 pass samples shows several large cracks from the surface going through the deposit, along with widespread cracking at interparticle bonds. It is clear that the thermal cycling as a result of 10 passes of the laser has fundamentally compromised the structure of the deposit.



(a) As sprayed (b) Heat treated, 1 pass



(c) Heat treated, 5 passes (d) Heat treated, 10 passes

Figure 7: Cross sections of mounted and polished stainless steel samples after laser heat treatment

Titanium

Figure 8 shows the hardness results for titanium samples for a laser traverse speed of 10 mm/s. Similar to the stainless steel results, there is no clear reduction in hardness due to heat treatment, with the mean hardness being higher for the heat treated samples than for the as sprayed samples. This would indicate the formation of a surface oxide layer [16].

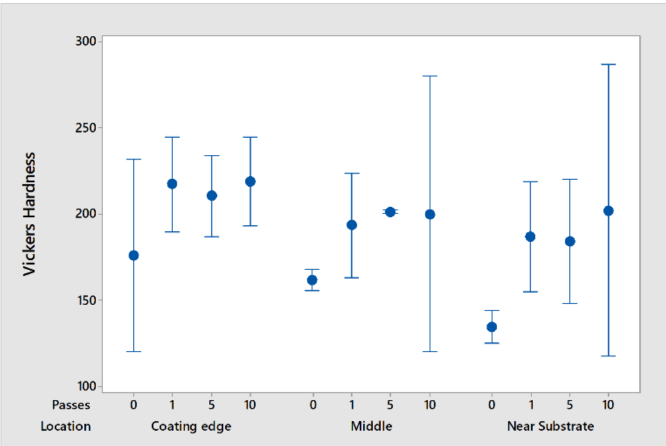
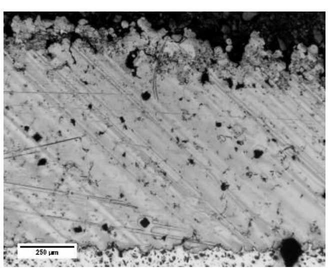
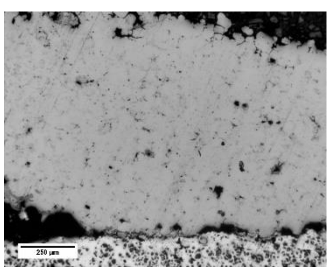


Figure 8: Coating hardness results for titanium samples. Error bars are 95% confidence intervals.

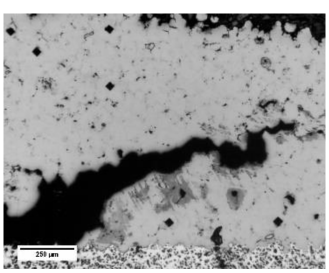
Figure ***9*** shows polished cross sections of the titanium samples. The 1 pass and 5 passes samples have significant cracking, with the 1 pass sample appearing to delaminate from the substrate, while the 5 passes sample having a large crack originating at the substrate and going through the deposit. The heat treatment process has caused significant damage to the structure of the deposit in each of these cases, with no discernable improvement in the properties of the material.



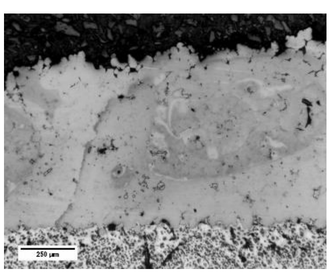
(a) As sprayed



(b) Heat treated, 1 pass



(c) Heat treated, 5 passes



(d) Heat treated, 10 passes

Figure 9: Cross sections of mounted and polished titanium samples after laser heat treatment

Conclusions

An investigation into the use of a CO_2 laser as an in process heat treatment for cold sprayed materials was carried out. From this work the following conclusions can be made:

- Laser heat treatment was shown to reduce the hardness of aluminium 6061 and copper samples, indicating that some of the residual stresses caused by stain hardening were relieved, without causing any changes in material structure or porosity.
- This effect was not seen for stainless steel or titanium samples, though the titanium samples did show evidence of the formation of a surface oxide layer.
- Laser heat treatment was shown to cause cracking within the deposit and at the substrate-deposit interface in some cases for stainless steel and titanium. Multiple passes of a laser heat treatment must be done carefully to avoid cracking from thermal cycling.
- The feasibility of using a CO_2 laser to heat treat single tracks of cold sprayed materials was shown.
- Further investigation is required to optimize the heat treatment process for each material, and to investigate the optimal strategy for implementing an in-process laser heat treatment system for cold spray.

Acknowledgments

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References

[1] Champagne, V., *The cold spray materials deposition process: Fundamentals and applications*. Elsevier, (2007).

[2] Luzin, V., Spencer, K., and Zhang, M. X., “Residual stress and thermo-mechanical properties of cold spray metal coatings,” *Acta Mater.*, Vol. 59, No. 3, (2011), pp. 1259–1270.

[3] Vardelle, A. *et al.*, “The 2016 Thermal Spray Roadmap,” *J. Therm. Spray Technol.*, Vol. 25, No. 8, (2016), pp. 1376–1440.

[4] Shayegan, G. *et al.*, “Residual stress induced by cold spray coating of magnesium AZ31B extrusion,” *Mater. Des.*, Vol. 60, (2014), pp. 72–84.

[5] Rech, S., Trentin, A., Vezzù, S., Legoux, J.-G., Irissou, E., and Guagliano, M., “Influence of Pre-Heated Al 6061 Substrate Temperature on the Residual Stresses of Multipass Al Coatings Deposited by Cold Spray,” *J. Therm. Spray Technol.*, Vol. 20, No. 1–2, (2011), pp. 243–251.

[6] Li, W., Yang, K., Zhang, D., and Zhou, X., “Residual Stress Analysis of Cold-Sprayed Copper Coatings by

Numerical Simulation," *J. Therm. Spray Technol.*, Vol. 25, No. 1–2, (2016), pp. 131–142.

[7] Suhonen, T., Varis, T., Dosta, S., Torrell, M., and Guilemany, J. M., "Residual stress development in cold sprayed Al, Cu and Ti coatings," *Acta Mater.*, Vol. 61, No. 17, (2013), pp. 6329–6337.

[8] Al-Mangour, B., Dallala, R., Zhim, F., Mongrain, R., and Yue, S., "Fatigue behavior of annealed cold-sprayed 316L stainless steel coating for biomedical applications," *Mater. Lett.*, Vol. 91, (2013), pp. 352–355.

[9] Moridi, A., Hassani-Gangaraj, S. M., Vezzù, S., and Guagliano, M., "The effect of (severe) shot peening as pre/post treatment on fatigue behavior of cold spray coating," *Proc. Int. Therm. Spray Conf.*, Vol. 283, (2014), pp. 611–616.

[10] Lee, C. and Kim, J., "Microstructure of Kinetic Spray Coatings: A Review," *J. Therm. Spray Technol.*, Vol. 24, No. 4, (2015), pp. 592–610.

[11] Hall, a. C., Cook, D. J., Neiser, R. a., Roemer, T. J., and Hirschfeld, D. a., "The Effect of a Simple Annealing Heat Treatment on the Mechanical Properties of Cold-Sprayed Aluminum," *J. Therm. Spray Technol.*, Vol. 15, No. 2, (2006), pp. 233–238.

[12] Kumar, S., Jyothirmayi, A., Wasekar, N., and Joshi, S. V., "Influence of annealing on mechanical and electrochemical properties of cold sprayed niobium coatings," *Surf. Coatings Technol.*, Vol. 296, (2016), pp. 124–135.

[13] Rokni, M. R., Widener, C. a., Champagne, V. K., and Crawford, G. a., "Microstructure and mechanical properties of cold sprayed 7075 deposition during non-isothermal annealing," *Surf. Coatings Technol.*, Vol. 276, (2015), pp. 305–315.

[14] MacDonald, D., Fernández, R., Delloro, F., and Jodoin, B., "Cold Spraying of Armstrong Process Titanium Powder for Additive Manufacturing," *J. Therm. Spray Technol.*, Vol. 26, No. 4, (2017), pp. 598–609.

[15] Lupoi, R., Sparkes, M., Cockburn, A., and O'Neill, W., "High speed titanium coatings by supersonic laser deposition," *Mater. Lett.*, Vol. 65, No. 21–22, (Nov. 2011), pp. 3205–3207.

[16] Carlone, P., Astarita, A., Rubino, F., Pasquino, N., and Aprea, P., "Selective Laser Treatment on Cold-Sprayed Titanium Coatings: Numerical Modeling and Experimental Analysis," *Metall. Mater. Trans. B*, Vol. 47, No. 6, (2016), pp. 3310–3317.

[17] Astarita, A., Genna, S., Leone, C., Minutolo, F. M. C., Rubino, F., and Squillace, A., "Study of the Laser Remelting of a Cold Sprayed Titanium Layer," *Procedia CIRP*, Vol. 33, (2015), pp. 452–457.

[18] Aldwell, B., Yin, S., Mcdonnell, K. A., Trimble, D., Hussain, T., and Lupoi, R., "A novel method for metal – diamond composite coating deposition with cold spray and formation mechanism," *Scr. Mater.*, Vol. 115, (2016), pp. 10–13.

[19] Kumar, S., Vidyasagar, V., Jyothirmayi, A., and Joshi, S. V., "Effect of Heat Treatment on Mechanical Properties and Corrosion Performance of Cold-Sprayed Tantalum Coatings," *J. Therm. Spray Technol.*, (2016).