

Hybrid additive manufacture of 316L stainless steel with cold spray and selective laser melting: microstructure, mechanical properties and case study

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Abstract

A novel hybrid additive manufacturing process was proposed and utilized for the production of 316L stainless steel components in this work. It combines selective laser melting (SLM) and cold spraying (CS), allowing the fabrication of complex structures with SLM and the rapid manufacture of simple features with CS. The underlying principle of the hybrid additive manufacturing is to use CS to deposit a 316L stainless steel structure onto an SLM 316L stainless steel component, followed by heat treatment and finish machining. The microstructure and mechanical properties of the as-fabricated and heat-treated CS/SLM part, and the CS/SLM interfacial bonding features were studied. In the as-fabricated state, the CS part has a dendritic structure similar to the feedstock, while the SLM part is characterized by cellular subgrains confined in coarse grain structures. Due to recrystallisation after heat treatment, the definition of interparticle boundaries diminished, equiaxed coarse grains and twinning were formed, and the extremely fine cellular subgrains are removed from the SLM part. Due to the ‘fusion’ nature of the process, the SLM sample delivered improved mechanical properties when compared to the CS sample, even after heat treatment which significantly improves its mechanical properties. Heat treatment also improves the interfacial bond strength between the CS part and SLM part due to enhanced atomic diffusion. The case study demonstrates that the proposed hybrid additive manufacturing is a promising technique for the manufacture of free-standing components, modification of fabricated components and the repair of damaged components.

Introduction

Selective laser melting (SLM) is an emerging laser additive manufacturing technology, which is used for producing metallic, metal matrix composite and ceramic components. The manufacture process relies on a high-power laser beam selectively melting the powders in a powder bed layer by layer. The liquid melt pool created by the laser then cools down and solidifies rapidly to form a solid track which when combined with adjacent tracks and layers, forms components (1). With the integrated high-precision diagnosis and image processing techniques, SLM technology can be applied to manufacture components with very complex structures and fine details. As

compared with other conventional subtractive manufacturing processes, SLM allows free design of component structure, rapid manufacture of components in high spatial resolution, and customization of components in small-scale production (1–5). The mechanical properties of the SLM components are found to be nearly equivalent with the conventionally manufactured counterparts. Due to these superior advantages, SLM has been well recognized as the technology of a future manufacturing industry. However, since the laser melting process is time consuming, building a SLM component generally needs long production times.

In comparison to the fusion-based SLM process, cold spraying (CS) is a solid-state material deposition and additive manufacturing technology (6,7). In this process, high-temperature compressed gases (typically nitrogen and helium) are used as the propulsive gas to accelerate microscale metal powder feedstock to a high velocity. The high-velocity particles impact onto a substrate and then consolidate into a bulk deposit at a temperature well below the melting temperature to realize the additive manufacture of a part or feature (8). The consolidation of a CS deposit relies on particle kinetic energy prior to impact and the consequent severe plastic deformation of powder feedstock rather than melting and re-solidification. Therefore, CS offers many unique advantages that fusion-based additive manufacturing processes do not have, such as lower thermal effect, no phase transformation, high production efficiency, free design of component size and damaged component restoration (9,10). However, CS deposits typically have rough contour and surface in their as-fabricated state since CS is not a precise manufacturing technology (11).

316L stainless steel has been widely used in medical implants and food industries due to its excellent corrosion-resistance. It is also a good candidate alloy for SLM and CS due to its low reflectivity, good wettability and ductility (1,2). A number of works have been done to investigate the manufacturing parameters, microstructures and properties of SLM and CS 316L stainless steel (6,12–25). The referenced studies demonstrate that high-density 316L stainless steel with excellent mechanical properties can be produced through SLM using optimized laser parameters and scanning strategies (20). Due to the high cooling rate and rapid solidification during the manufacturing process, the SLM 316L stainless steel typically has a fine grain structure in the as-fabricated state (6,12,13,17,19–22,25), and thus demonstrates higher yield strength, tensile strength and hardness as compared with

wrought and cast counterparts (19,22,26). Heat treatment was found to result in the growth of cellular structures of the SLM 316L stainless steel and therefore the reduction of strength (15,16). As for CS, it has been shown that dense 316L stainless steel can also be produced with CS (27,28). However, due to the lack of sufficient metallurgical inter-particle bonding, the properties of as-fabricated CS 316L stainless steel are generally lower as compared with bulk counterparts (28–31). Heat treatment is capable of improving the properties of 316L stainless steel to close to that of bulk counterparts due to the mitigation of inherent defects (porosity and inter-particle boundaries) (28,31,32).

As addressed above, SLM and CS have unique advantages. The fusion-based SLM process is robust in producing complex steel parts with fine details but not in a time efficient manner, while solid-state CS process builds components rapidly but lacks manufacturing accuracy (7). Hence, the primary objective of the present paper is to combine both additive manufacturing technologies as a hybrid manufacturing process. The objective is to take advantage of the benefits of each process, using SLM to produce complex or detailed structures and then using CS to produce simple structures to reduce production time. As a preliminary study, 316L stainless steel was chosen as the material used in this study due to its suitability for both SLM and CS processes. Figure 1 shows the flowchart of the manufacturing procedure of the hybrid additive manufacturing process proposed in this work. Firstly, a 316L stainless steel part is manufactured by SLM; secondly, another 316L stainless steel part is deposited onto the SLM part using CS; thirdly, the as-fabricated CS/SLM part is heat treated in a furnace in order to improve the property of the CS part and the interfacial bond strength between the CS and SLM parts; finally, the heat-treated CS/SLM part is machined to provide an acceptable finish. Following the manufacture, microstructure and mechanical properties of the CS/SLM 316L stainless steel part before and after heat treatment will be studied. Investigation of the interfacial bonding features between the CS and SLM parts will also be conducted. In addition, a case study of using CS 316L stainless steel to repair a damaged SLM 316L stainless steel part was also carried out to prove the feasibility of CS process for modifying and repairing SLM parts.

2. Experimental methodology

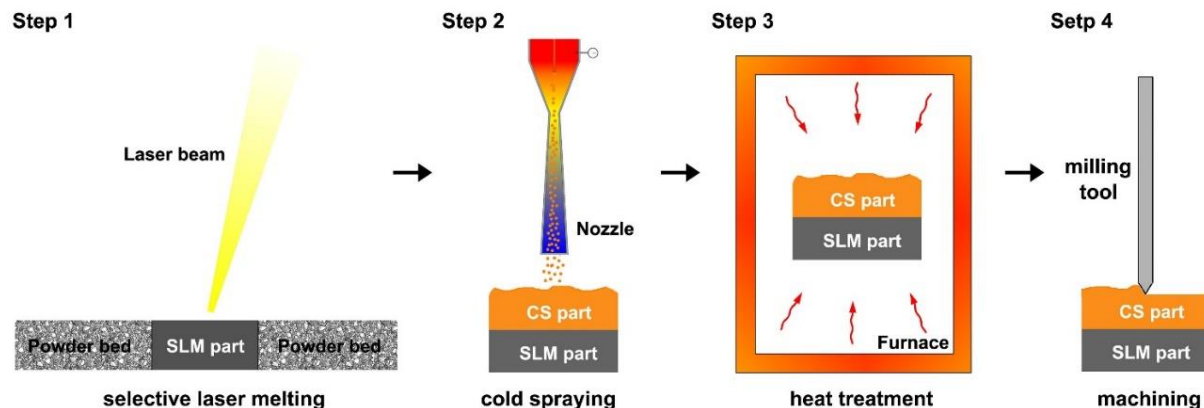


Figure 1: Flowchart of the manufacturing procedure of the hybrid additive manufacturing process proposed in this work

2.1. Manufacturing procedure

Spherical 316L stainless steel powder (EOS GmbH, Germany) with a size range of between 33 and 40 μm was used as the feedstock for SLM. Figure 2a shows the surface morphology of the 316L stainless steel powder observed using an SEM (Carl Zeiss ULTRA, Germany). EOS M290 SLM system (EOS GmbH, Germany) was used to manufacture the 316L stainless steel parts. Manufacture was performed under the argon environment with the substrate preheated to a temperature of 80 $^{\circ}\text{C}$. The scanning parameters were optimised by the orthogonal experiment method (33), and the laser scanning trajectory follows a zigzag pattern with an angle of 67 $^{\circ}$ between adjacent layers. Table 1 lists the scanning parameters used in this study. For CS process, the 316L stainless steel powders (LPW Technology Limited, UK) also have a spherical shape with the size range of between 15 and 45 μm . Figure 2b shows the surface morphology of the 316L stainless steel powders used for CS. The etched cross-section of a 316L stainless steel powder is also provided as an insert in Fig. 2b where the grains are characterised by multi-crystalline dendritic structure. The CS 316L stainless steel part was deposited onto the SLM 316L stainless steel part using an in-house CS system (Trinity College Dublin, Ireland) (34). Nitrogen and helium were used as the propulsive gases in this work as detailed in Table 1 which provides the working parameters for the CS process. Following the manufacture of the CS/SLM 316L stainless steel part, heat treatment was performed in air at a temperature of 1000 $^{\circ}\text{C}$ for 4 hours.

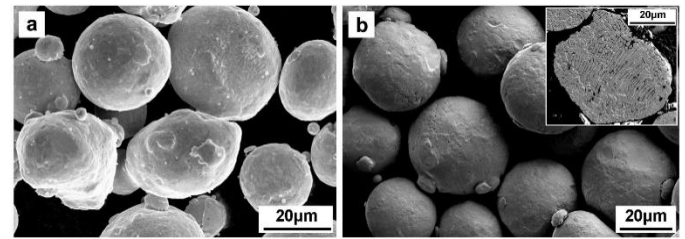


Figure 2: Surface morphology of the powders used in the experiments: (a) 316L stainless steel powders for SLM, and (b) 316L stainless steel powders for CS with the etched cross-section of a single powder inserted.

Table 1: Experimental parameters of the SLM and CS processes used in this work

SLM	Laser spot diameter	Laser power	Layer thickness	Hatch distance	Laser beam moving speed	Chamber environment
	100 μm	195 W	50 μm	90 μm	1.0 m/s	Vacuum (Ar)
CS	Pressure	Temperature	Standoff distance	Hatch distance	Nozzle moving speed	Propulsive gas
	3.0 MPa	1000 $^{\circ}\text{C}$	30 mm	3.5 mm	50 mm/s	N_2
	3.0 MPa	25 $^{\circ}\text{C}$	30 mm	3.5 mm	100 mm/s	He

2.3. Materials characterisation

The cross-sectional microstructures of the SLM part, CS part and the CS/SLM interface were studied by SEM. The samples were polished using standard metallographic procedures with the final polish applied using 0.06 μm colloidal silica solution for accessing the microstructure analysis. Polished samples were electrochemically etched using a reagent of 69% nitric acid and 31% water while applying 2 V DC current to observe and study the grain structure. Electron backscatter diffraction (EBSD) was also used to characterize the grain structure at the CS/SLM interfacial region in high magnification. Energy dispersive X-ray spectroscopy (EDX) was employed to study the oxidation behaviour of the heat-treated CS part. The porosity of the CS and SLM parts was measured using binary image analysis method with an open source image analysis software (Image J). The cross-sectional images were converted into a binary format, and then the porosity is calculated as the area ratio between the black pores and white surface background. Five micrographs were captured for each sample, and the averaged value was considered the sample porosity.

2.4. Mechanical property tests

The microhardness of the SLM and CS parts before and after heat treatment were tested using a Vickers hardness indenter (Mitutoyo, Japan) with a load of 500 g and dwell time of 10 s. Ten locations were tested on each sample, and the average value was considered the sample microhardness. The tensile strength of the SLM and CS parts was measured using a universal tensile strength system (Instron 8801, UK) at a displacement rate of 2 mm/min. The SLM tensile specimens were produced in a dog-bone shape by SLM directly with a gauge length of 25 mm, gauge width of 5 mm and thickness of 2.2 mm. The CS tensile specimens were machined into the same-size dog-bone shape from the as-fabricated deposit. The ultimate tensile strength (UTS) and break elongation (EL) before and after heat treatment were determined based on the average value of three tensile specimens. The surface morphologies of the fractured tensile specimens were then studied by SEM. The interfacial bond strength between the CS and SLM parts was measured based on the standard pull-off test (ASTM C-633-01). To prepare the test specimens, CS 316L stainless steel was first deposited onto a round-shape SLM 316L stainless steel substrate with a diameter of 25 mm using helium as the propulsive gas (Table 1). Then, the commercially available adhesive glue (Sader, France) with a maximum tensile strength of 70 MPa was used to attach the test specimens to the platen. The assembled specimens were tested by the tensile tester (IC ESCOFFIER, Estotest 50, France) with a cross-head speed of

1.56 mm/min. The fracture surfaces were then studied using SEM.

3. Results and discussion

3.1. Microstructure of the CS and SLM 316L stainless steel parts

Figure 3 shows the cross-sectional images of the CS and SLM parts before and after heat treatment. It is clear that the SLM part is denser than the CS parts in both as-fabricated and heat-treated states, and using helium as the propulsive gas results in denser deposit than using nitrogen due to the improved particle plastic deformation upon impact. The measured porosity shown in Fig. 4 further confirms this observation. However, after heat treatment, the inter-particle boundaries of the CS parts became much more visible than in the as-fabricated state. To explain this phenomenon, Fig. 5 shows the cross-sectional image of the nitrogen-produced CS part under high magnification and the corresponding EDX line scan at the interparticle boundary area. The EDX result indicates that the visible inclusions in the interparticle boundaries are the oxides of Cr and Mn. These oxides are likely to be Mn_2CrO_4 , FeCr_2O_4 and Cr_2O_3 , which are typical oxides of 316L stainless steel when exposed to air (35). The formation of the oxides may be due to the air penetrating into the deposit through poor-bond interparticle boundaries during annealing. The oxides in the interparticle boundaries act as a barrier to ‘sintering’, reducing the metallurgical bond strength between particles and leading to less favorable mechanical properties.

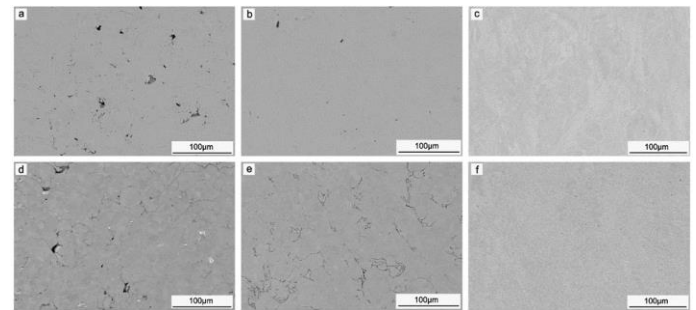


Figure 3: Cross-sectional images of the CS and SLM parts before and after heat treatment: (a) CS (N_2)-AF, (b) CS (He)-AF, (c) SLM-AF, (d) CS (N_2)-HT, (e) CS (He)-HT and (f) SLM-HT. ‘AF’ means as-fabricated, ‘HT’ means heat-treated.

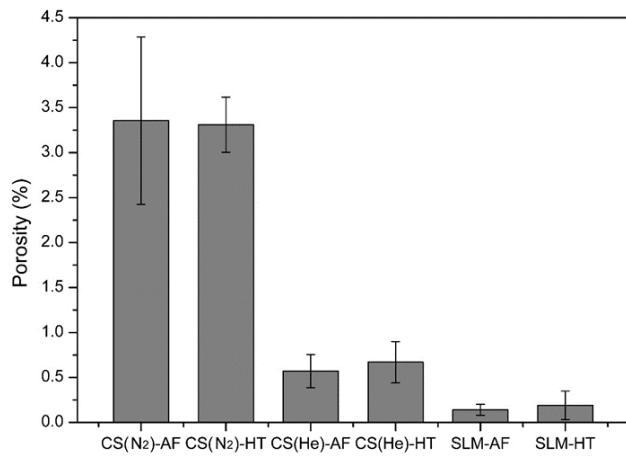


Figure 4: Porosity measurement of the CS and SLM 316L stainless steel parts before and after heat treatment

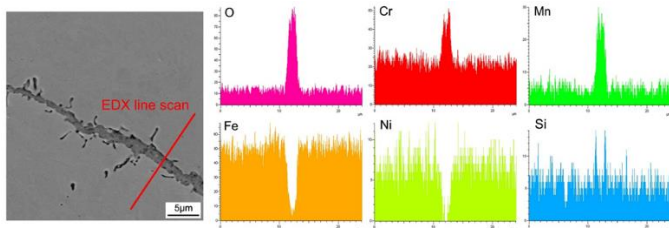


Figure 5: Cross-sectional image of the nitrogen-produced CS part under high magnification and the corresponding EDX line scan at the interparticle boundary area

Figure 6 shows the etched cross-sectional images of the CS and SLM parts before and after heat treatment. There is a significant difference between the grain structure of the CS parts and SLM part. For the CS parts in the as-fabricated state, inter-particle boundaries and grain boundaries are visible after etching. The etching is more prominent at the inter-particle boundaries than at the grain boundaries, which indicates that the inter-particle boundaries are the weakest point of the CS deposit. The grain structure of the deposited CS particles is similar to that of the original feedstock but exhibits obvious signs of plastic deformation. There is no significant difference in grain structure between the nitrogen-produced and helium-produced CS parts in the given images. However, the grain structure of the as-fabricated SLM part is very different from that of the CS parts. The white dotted lines shown in Fig. 6c indicate the grain boundaries, and it is clear that the grain size of the as-fabricated SLM part is much larger than that of the CS part. In addition, within each grain, very fine cellular subgrains with an intercellular spacing of hundreds of nanometers were formed as can be seen from the insert figure in Fig. 6c. The formation of the fine subgrains is due to the extremely high cooling rate of the molten pool which prevents the development of secondary dendrite arms during the solidification process (25). The orientation of cellular subgrains was not uniform across all grains. These fine cellular subgrains can lead to an improvement of the hardness and strength of SLM steels as compared with conventionally manufactured steels (19,22,26,36).

After heat treatment, the microstructure of the CS parts and SLM parts exhibited different characteristics when compared to

the as-fabricated state. For the CS parts, recrystallisation took place both inside the particles and across the inter-particle boundaries. This results in the growth of grains and the 'sintering' of the inter-particle boundaries through atomic diffusion (37). As a consequence, the microstructure is characterised by less well defined interparticle boundaries with equiaxed coarse grains and twinning. When comparing the nitrogen-produced CS part to the helium-produced one, the former has more oxide inclusion in the inter-particle boundaries than the latter. This may be due to the weaker interparticle bonding and higher porosity of the nitrogen-produced CS part. For the SLM part, extremely fine cellular subgrains are removed after heat treatment due to the recrystallisation (16).

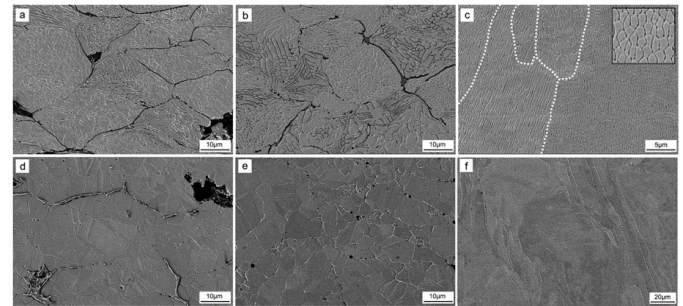


Figure 6: Etched cross-sectional images of the CS and SLM parts before and after heat treatment: (a) CS (N2)-AF, (b) CS (He)-AF, (c) SLM-AF, (d) CS (N2)-HT, (e) CS (He)-HT and (f) SLM-HT. 'AF' means as-fabricated, 'HT' means heat-treated. White dotted lines indicate the grain boundaries.

3.2. Mechanical properties of the CS and SLM 316L stainless steel parts

Figure 7 shows the microhardness of the CS parts and SLM part before and after heat treatment. It is evident that the CS parts have a higher hardness than the SLM part. This is due to the occurrence of deformation-induced work-hardening during the CS deposition. The helium-produced CS part is even harder than the nitrogen-produced one due to the more prominent plastic deformation which occurs due to the higher particle impact velocity. After heat treatment, the hardness of the CS parts and SLM part reduces due to the recrystallisation and resulting grain growth.

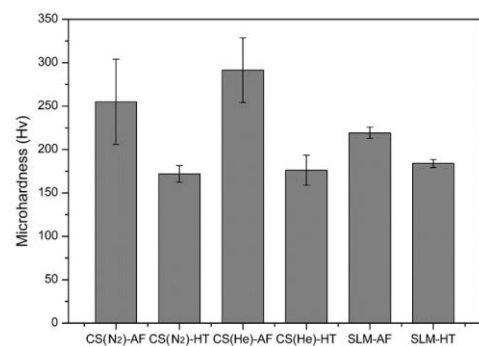


Figure 7: Microhardness of the CS and SLM 316L stainless steel parts before and after heat treatment

Figure 8 shows the UTS and EL of the CS and SLM parts before and after heat treatment. For comparison, the UTS and EL of

wrought 316L stainless steel are also included in Fig. 8. As expected, the CS parts had lower UTS and EL as compared with the SLM part and wrought 316L stainless steel due to the inherent defects (pores and inter-particle boundaries) in the CS parts (38). After annealing, both the UTS and EL of the CS parts increase due to the improved inter-particle bonding and the occurrence of recrystallisation in the CS deposits [19,20,41]. The helium-produced CS part has higher UTS and EL than the nitrogen-produced part in both as-fabricated and heat-treated states due to the improved inter-particle bonding and lower porosity. In contrast with the CS parts, the SLM part and wrought 316L stainless steel underwent improvement in ductility but sacrificed UTS due to the recrystallisation and consequential grain growth. The fractographic images of the tensile specimens of the CS and SLM parts before and after heat treatment are shown in Fig. 9. For the CS parts, the fracture surfaces of the as-fabricated specimens reveal inter-particle failure which is a clear indication of the occurrence of brittle fracture, while the fracture surfaces of the heat-treated specimens are characterised by both inter-particle failure and dimple-like features. The presence of dimple-like features is evidence of high-quality cohesion strength and high ductility (38). For the SLM part, in the as-fabricated state, the fracture surface is characterised by mainly dimple-like features with some cleavage-like features. However, in the heat-treated state, the fracture surface reveals only dimple-like features, which explains why the ductility of the heat-treated SLM part has been enhanced.

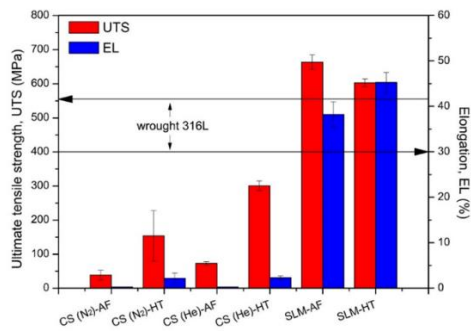


Figure 8: UTS and EL of the CS and SLM 316L stainless steel parts before and after heat treatment.

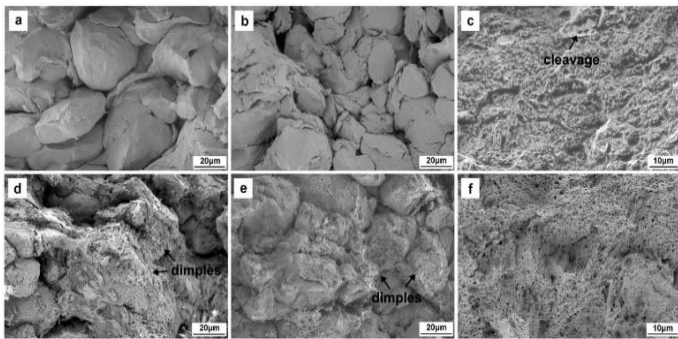


Figure 9: Fractographic images of the CS and SLM tensile specimens before and after heat treatment: (a) CS (N₂)-AF, (b) CS (He)-AF, (c) SLM-AF, (d) CS (N₂)-HT, (e) CS (He)-HT and (f) SLM-HT. 'AF' means as-fabricated, 'HT' means heat-treated.

3.3. Interfacial bonding features between the CS and SLM 316L stainless steel parts

The interfacial bonding of the CS/SLM part is one of the most important factors that determines the potential of the hybrid additive manufacturing process proposed in this work. Therefore, it is necessary to investigate the interfacial bonding strength between the CS part and SLM part. In Section 3.2, the tensile tests concluded that using helium as the propulsive gas results in improved mechanical properties compared to nitrogen. Therefore, in this section, to maximize the mechanical properties of the CS/SLM part, only helium-produced CS/SLM parts are studied. Fig. 10 shows the cross-sectional images and etched cross-sectional images of the CS/SLM part before and after heat treatment. It is evident that the interface of the as-fabricated part is clean, smooth and is not clearly defined. However, after heat treatment, sections of the interface become visible due to the formation of oxides. After etching, a drastic change in grain structure can be observed at the as-fabricated CS/SLM interface. For the heat-treated part, the grain structure change is less prominent due to the recrystallisation in the CS and SLM parts. In addition, it is also interesting to find that the grain size of the SLM part near the interface is smaller than further down in the sample, which may arise from the grain refinement caused by the severe plastic deformation during the impact of CS SS 316L particles (40).

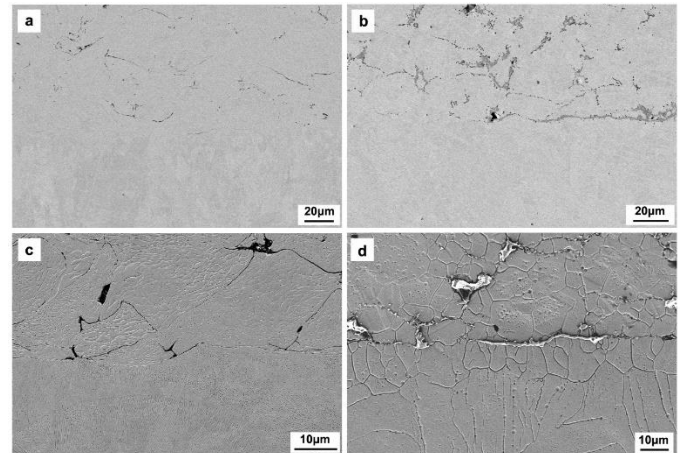


Figure 10: Cross-sectional images and etched cross-sectional images of the CS/SLM part before and after heat treatment: (a) CS/SLM-AF, (b) CS/SLM-HT, (c) etched CS/SLM-AF and (d) etched CS/SLM-HT. 'AF' means as-fabricated, 'HT' means heat-treated.

Figure 11 shows the interfacial bond strength of the CS/SLM part before and after heat treatment. The interfacial bond strength was measured to be 22.02 ± 2.93 and 51.48 ± 0.88 MPa for the as-fabricated and heat-treated specimens, respectively. Note that all the as-fabricated specimens underwent adhesive failure, while all the heat-treated specimens delaminated from the glue, which means their actual interfacial bond strength is higher than 51.48 ± 0.88 MPa. Therefore, heat treatment significantly improves the interfacial bond strength by more than 2.5 times. The higher interfacial bond strength of the heat-

treated part is due to the enhanced atomic diffusion and recrystallisation across the interface of the CS and SLM parts, which is confirmed by the EBSD image at the CS/SLM interfacial region shown in Fig. 12. It is clear that recrystallisation took place across the CS/SLM interface and thus the no clear interface can be observed in Fig. 12.

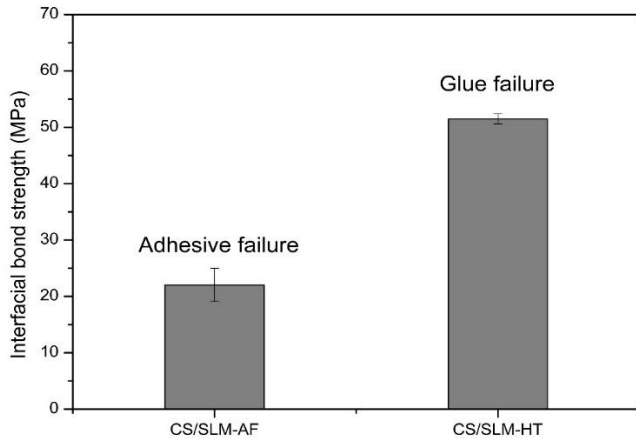


Figure 11: The interfacial bond strength of the CS/SLM part before and after heat treatment. 'AF' means as-fabricated, 'HT' means heat-treated.

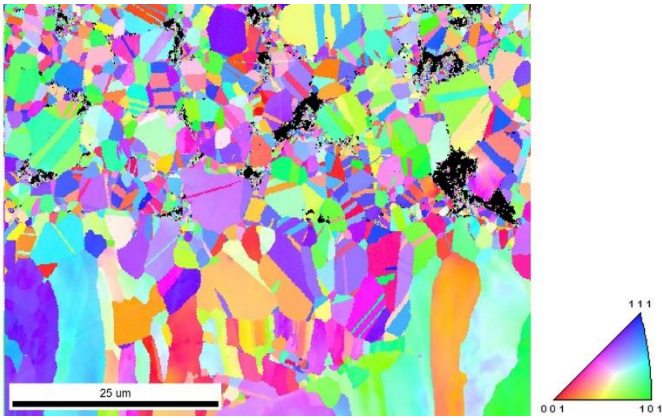


Figure 12: EBSD image at the CS/SLM interfacial region after heat treatment. The black colour indicates oxides.

3.4. Case studies

Two case studies were carried out to demonstrate the feasibility of the proposed hybrid additive manufacturing process in this work. The first is a free-standing CS/SLM 316L stainless steel part with a diameter of 25 mm and height of 7.5 mm. The part was manufactured following the procedure shown in Fig. 1 and manufacturing parameters shown in Table 1 with helium. CS SS 316L stainless steel was deposited onto an SLM 316L stainless steel substrate, and then heat-treated at 1000 °C for 4h in air, followed by machining. Fig. 13 shows the photos of the CS/SLM 316L stainless steel part manufactured from as-fabricated state to final machined state. The machined part shows strong interfacial bonding with a good finish, which indicates the feasibility of the proposed hybrid additive manufacturing process.



Figure 13: Photos of the CS/SLM 316L stainless steel part manufactured from as-fabricated state to final machined state

The other case is using CS 316L stainless steel to repair a damaged SLM 316L stainless steel part. Fig. 14 shows the photo of the repair process. The damaged area is a crater with a diameter of 20 mm and depth of 5 mm which was machined through milling. Following the CS deposition, the as-repaired part was heat-treated at 1000 °C for 4h and then finish machined. It is evident from Fig. 15 that the damaged SLM part was successfully repaired with an excellent finish using CS. This further highlights the novelty of the hybrid additive manufacturing process proposed in this work.

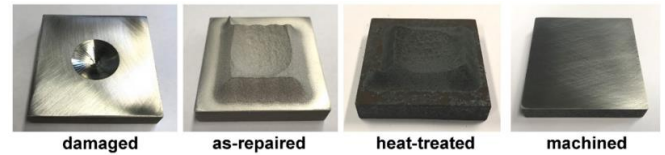


Figure 14: Photos of the repair process of a damaged SLM SS316L stainless steel part repaired with CS 316L stainless steel

Conclusions

In this work, a hybrid additive manufacturing process, combining cold spraying (CS) and selective laser melting (SLM), was proposed and used to produce 316L stainless steel components. The hybrid additive manufacturing process allows the fabrication of complex structures with SLM and simple structures with CS but much more rapidly. Based on the experimental results and discussion, the following conclusions were drawn:

1. The SLM part has lower porosity and higher tensile strength than the CS parts due to the 'fusion' nature of SLM; the helium-produced CS part has improved mechanical properties when compared to the nitrogen-produced component due to the enhanced inter-particle metallurgical bonding obtained at higher impact velocities.
2. The SLM part and CS parts have different grain structures. The grain structure of the CS part has no discernible difference from that of the original feedstock, while the SLM part is characterised by fine cellular subgrains with an intercellular spacing of hundreds of nanometres confined in coarse grains.
3. Heat treatment results in the occurrence of recrystallisation in the CS and SLM parts. As a consequence, the microstructure of the CS part has less well defined interparticle boundaries, equiaxed coarse grains and twinning, and the extremely fine cellular subgrains in the as-fabricated SLM part are enlarged.

4. Heat treatment significantly improves the tensile strength of the CS part and the interfacial bond strength between the CS part and SLM part due to the reduced inherent defects and enhanced atomic diffusion.
5. The case study demonstrates the feasibility and potential of the hybrid additive manufacturing for the production of free-standing parts, modification of fabricated components and the repair of damaged parts.

Acknowledgments

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