Properties for AA7050 Deposits Produced by the Cold Spray Process

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ABSTRACT

The presentation describes the microstructure, adhesion and shear test results for AA7050 deposited AA7050-T7451 substrates via the Cold Spray process using helium and nitrogen as the main process gas. A batch of gas atomized AA7050 powder sized to -270 mesh (<53 μm) was sieved to +625 (>20μm) to remove as many of the smaller particles as possible. A portion of the powder was thermally treated using a propriety process. Particle size distribution measurements were made for the powders in the as-received, sieved and sieve and thermally treated powders. Scanning electron images of the powder before and after the thermal treatment were taken. The AA7050 powder was deposited on AA7050-T7451 substrates in the as-received and as-received and thermally treated conditions. Microstructure analysis showed deformation characteristics of the powders. Deposits of all the powder/main gas combinations showed very good adhesion. The sear strength for the AA7050 deposited using helium was much higher than the shear strength of the samples produced using nitrogen. Adhesion and shear strengths produced using a hand held spray nozzle and a robot controlled spray nozzle were similar.

1. INTRODUCTION

The Cold Spray process has been used to deposit a number of different alloys and has achieved the greatest success depositing aluminum (Al) alloys for nonstructural repair and dimensional restoration [1]. In the Cold Spray process, powder particles are injected into a high pressure gas stream that has been heated to below the melting temperature of the powder. The gas expands through a converging-diverging nozzle to supersonic conditions. The expanding gas accelerates the particles to speeds as high as 1,000 m/s. The particles impact a substrate and undergo severe plastic deformation which, under the right conditions, creates a bond between the particle and the substrate or previously deposited powder [1]. The deposits can have excellent adhesion strength and tensile strength but historically have had low ductility. Recent advancements in Cold Spray equipment, nozzle design and material have made it possible to increase the ductility in Al alloys [2]. The increased ductility has made it possible to further develop structural repair processes for Al alloys.

Deformation of the particles is an essential factor that governs the adhesion strength and mechanical properties of the deposited materials. The deformation is a function of particle materials properties such as melting temperature, tensile strength and density. Other factors that influence deposition include particle diameter, deposition temperature and impact velocity [3]. The tensile strength of the powder can be modified through thermal treatments. Particle size and particle size distribution can be controlled by sieving or air classifying. The impact velocity and particle deposition temperature are affected by the pressure, temperature and type of the main process gas and the nozzle geometry. Helium (He) has a much higher speed of sound that

nitrogen (N2) which results in a much higher gas velocity as the gas expands through the nozzle. Higher gas velocities result in greater drag between the gas and the powder particle and higher particle velocities. Helium is also more than 20 times as expensive as nitrogen. The use of helium improves the deposition efficiency (DE) which reduces the time and the amount of gas used during the process. The increase in DE becomes more important as the cost of the powder increases. Thermal treatments can improve the deposition efficiency and improved microstructure [4].

Although, structural repairs have been demonstrated as a doubler repair on the F/A-18 aircraft, additional work is needed to increase the ductility and fatigue strength before Cold Spray structural repair can be widely implemented [5]. The work presented in this paper was performed to gain a better understanding of the effects of main process gas and powder processing on the deposition characteristics of AA7050 powder on AA7050-T7451 substrates.

2. EXPERIMENTATION

2.1 Powder Processing

A batch of -270 mesh (<53 μm) AA7050 powders was purchased from Valimet Inc., Stockton, CA. The powder was sieved through a screen to +635 mesh (>20μm) to remove as many of the fine powder particles as possible. A portion of the powder was thermally treated using a propriety process. Particle Size Distribution (PSD) measurement were performed on sieved and on sieved and thermally treated powders using a Malvern Panalytical Mastersizer 3000. This device uses dry dispersion laser diffraction to obtain a particle size distribution. Scanning Electron Microscope (SEM) images were taken of both the as-received and thermally processed AA7050 powders using a Quanta Q250 SEM. AA7050 samples were analyzed using a Nikon Epiphot 200 inverted microscope to capture micrograph images, measure coating thickness, and measure coating porosity. Using NIS Elements imaging software, porosity percentage was measured and recorded.

2.2 Cold Spray Deposition

The AA7050 powder was deposited using a VRC Gen III Hybrid Additive/Subtractive High Pressure Cold Spray Manufacturing System. As-received and thermally treated powders were deposited using N2 and He as the main process gas. Coatings were deposited onto AA7050-T7451 substrates for the adhesion and shear testing. The substrate material was purchased from McMaster-Carr Supply Company. Each substrate was prepared by polishing one surface with a green Scotch-Brite pad. The surface was then cleaned using alcohol. Deposition was performed immediately after the surface was cleaned. Cold spray deposition was performed using a VRC PBI nozzle with dimensions of 2 mm x 6.3 mm x 170 mm. The cold spray parameters used to deposit the AA7050 with N2 and He are shown in Table 1. The deposition process was controlled by a robot for four adhesion trials and six shear strength trials. Two samples each for the adhesion and shear strength trials was produced using a hand-held spray nozzle. The gas

pressure, gas temperature and powder feed rate for the hand-held depositions were the same as the robot but the stand-off distance and the nozzle traverse speed varied with the movement of the operator.

Table 1. Cold spray deposition parameters.

Spray Gas	N_2	He
Gas Temperature	425 °C	450 °C
Gas Pressure	4.48-5.86 MPa	3.62 MPa
Powder Feed Rate	6.00 rpm	4.00 rpm
Standoff Distance	25.4 mm	25.4 mm
Nozzle Traverse Speed	200 mm/s	400 mm/s

2.3 Deposition Characterization

A PSD was performed on the as-received -270 mesh (53 micron), the sieved +635 mesh, and thermally treated powder that was sieved to +635 mesh. Powder from each variant was mounted and polished and examined using an SEM. After specimens were sprayed, they were sectioned and mounted using a Struers LaboPress-1 mounting device that encapsulated specimens in a phenolic mount. Specimens were then polished using a LECO GPX300 polisher. The AA7050 coated surface was etched with Keller's Reagent. The samples were examined using a Nikon Epiphot 200 inverted microscope. NIS Elements imaging software was used to determine the porosity and coating thickness.

Adhesion measurements were performed in accordance with ASTM D4541-17 Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Tester [6]. Approximately 0.9 mm of AA7050 powder was deposited onto AA7050-T7451 substrates with dimensions of 50.8 mm x 152.4 mm x 6.4 mm. The deposition was machined to a uniform thickness of 0.5 mm. Four, equally spaced areas were machined into the bond plate as shown in Figure 1. The outer diameter of the machined circle as 25.4 mm. A 10 mm diameter circle inside the 25.4 mm diameter circle remained after machining. Sufficient material was removed to expose the surface of the substrate. The mating surfaces of the 10 mm diameter circle and the 10 mm diameter pull specimen (dolly) were roughened with a red Scotch-Brite pad and cleaned with alcohol. Two disks of FM1000 adhesive purchased from Cytec Engineered Materials were applied to the 10 mm dolly mating site. A dolly slug was then placed on each of the four adhesion sites. A weight was placed on the dolly slugs and the substrate was placed in an oven heated to 230 º C for three hours. The substrate was removed and allowed to cool to room temperature before testing. Adhesion tests were performed using a DelFelsko AT Manual Dolly Pull-off Adhesion Portable Pump.

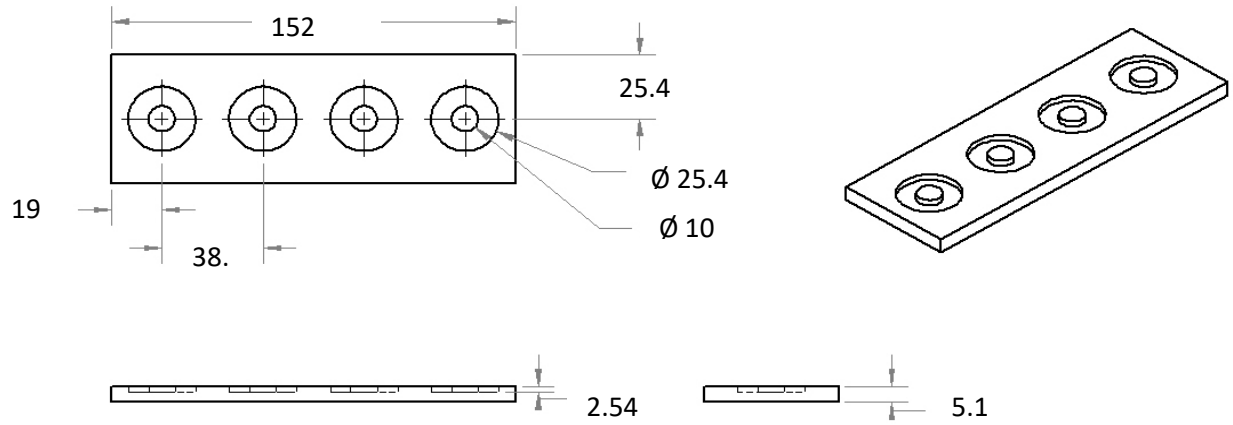


Figure 1. A schematic of the coated and machined substrate (top) and a photo of the dollies bonded to the coating prior to testing (bottom)

Shear testing was performed following MIL-J-29445 Triple Lug Shear Test [7]. AA7050 powder was deposited on an AA7050-T751 substrates with dimensions of 127 mm x 25.4 mm x 12.7 mm. The deposition was covered the middle 50.8 mm of the substrate at a thickness of approximately 4 mm. The deposition was machined to from two 20.3 mm x 5.0 mm x 3.2 mm lugs. The substrate was placed in a specially designed fixture and tested in a an Instorn. A picture of the test fixture and a schematic of the test samples are shown in Figure 2.

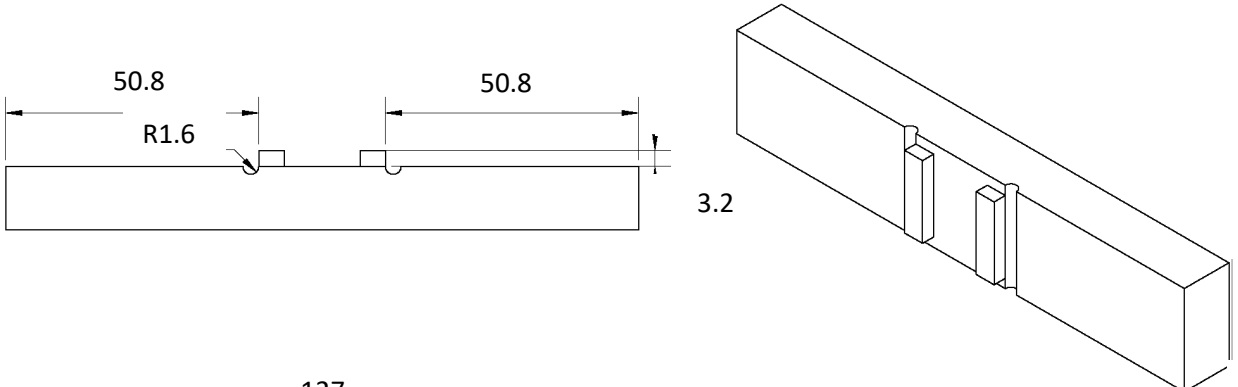


Figure 2. A schematic of the machined substrate (top) and a photograph of the shear test fixture and sample .

3. RESULTS

3.1 Powder Particle Size Distribution (PSD)

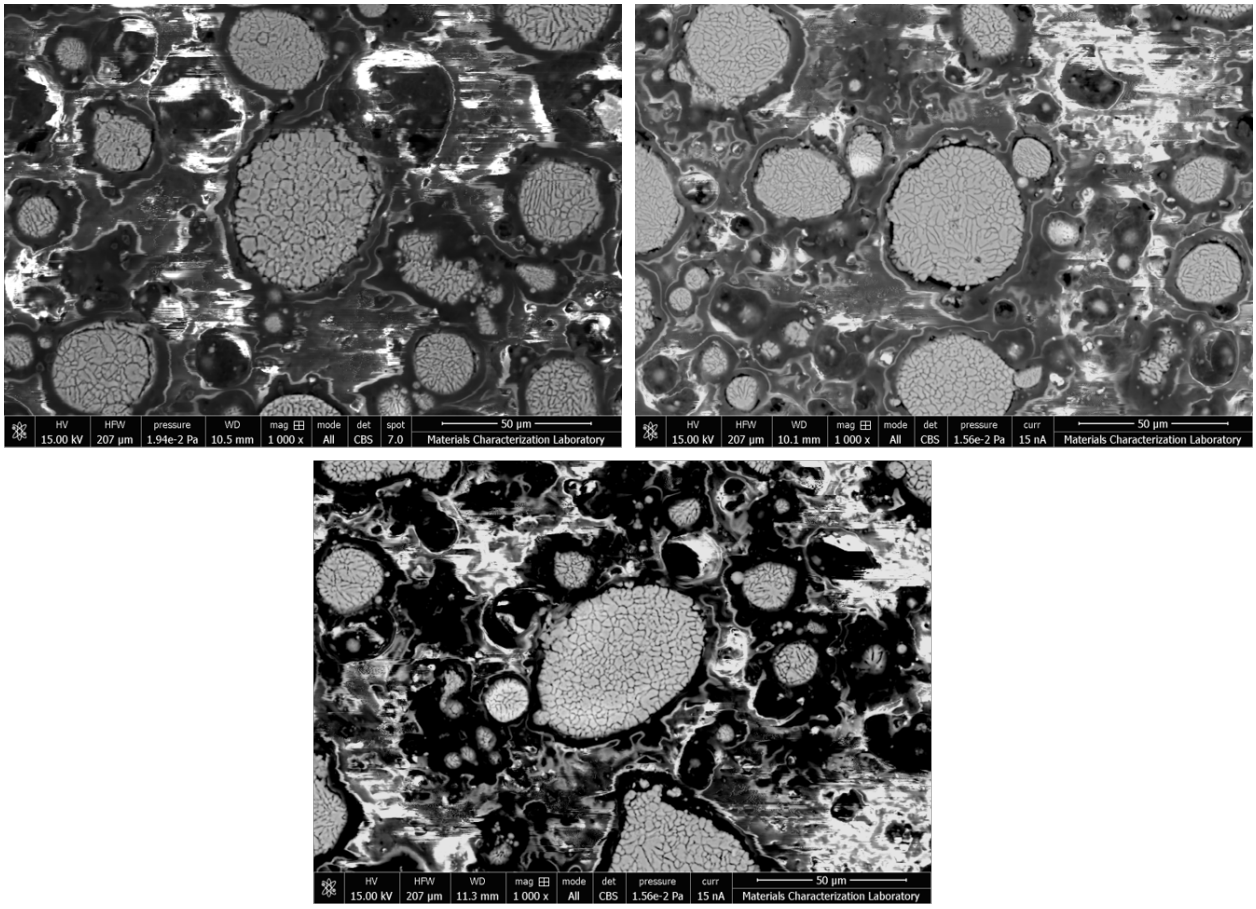
Particle size distribution measurements were taken for AA7050 as-received, sieved to +635 mesh and thermally treated sieved to +635 mesh powder prior to deposition trials. It was observed that all three powders shared similar particle size and average distribution. Powder particle sieving and thermal heat treatment did not change the particle size distribution indicating that the AA7050 has been sieved prior to shipment. PSD results showed the particles did not agglomerate during thermal treatment. The results of PSD testing are shown in Table 2.

Table 2. Particle Size Distributions of the AA7050 powder

Processing	D[3,2] µm	D[3,2] µm	D[3,2] µm	D[3,2] µm	D[3,2] µm
As-Received	12.8	24.7	6.18	21.7	47.6
Sieved to +635 mesh	14.3	25.2	7.20	22.6	46.8
Thermally Treated and Sieved	14.1	24.7	7.15	22.4	45.7

3.2 Powder Microstructure Analysis with Scanning Electron Microscope (SEM)

AA7050 in the as-received condition, in the sieved to +635 mesh and sieved and thermally treated powder was mount in epoxy for microstructural analysis. The powder samples were polished and etched to obtain SEM micrographs of the microstructure. The SEM images of the three powders are shown in Figure 3. The microstructures were typical of a gas atomized AA7050 powder. The temperature of the thermal treatment was not high enough to solutionize the powder or change the grain structure. Higher resolution analysis is needed to determine if any compositional changes occur during thermal treatment.



Fgure 3. SEM micrographs of (top left) as-received -270 mesh AA7050 Powder, (top right) sieved to +635 mesh AA7050 powder, (bottom middle) sieved to +635 mesh thermally treated AA7050 powder.

3.3 AA7050 Coating Microstructure

The microstructures from depositions produced for the as-received and the thermally treated powder applied using N2 and He were examined using optical microscopy. Specimens were

etched in order to see the grain structure and boundaries. The microstructures were examined to determine the effects of thermal treatment and carrier gas type on the deformation characteristics of AA7050 powder. The microstructures, show in Figure 4, have similar levels of deformation and porosity. There was greater deformation at the substrate-deposition interface for the samples produced with He. Depositions were much thicker when using He as the main process gas. The thickness per pass was greater which would tend to reduce the amount of deformation in the powder. This could be one reason why the deformations are similar for the He and N2 depositions. The process efficiency was measured for the powder/gas combinations. Process efficiency is the amount of powder that is deposited on the substrate divided by the total amount of powder sprayed during the deposition trial. This includes the time to nozzle is not over the substrate. Smaller samples have lower process efficiencies due to the amount of time the nozzle is off of the substrate. To remove the effect of samples size, the process efficiencies were divided by the lowest process efficiency to get a normalized deposition efficiency (NDE). The as-received powder deposited using N2 had the lowest process efficiency and was used as the baseline. Thermally treating the powder doubled the NDE when using N2 as the main process gas. The NDE for the as-received powder was 3.7 times higher than the baseline and the NDE for the thermally treated powder was 4.7 times higher when using He as the main process gas. NDE, coating thickness of average porosity results are shown in Table 3.

Table 3. Summary of average coating thickness, average coating porosity, and deposition efficiency.

Process Gas Type /Powder Treatment	**Average Coating Thickness (mm)**	**Average Coating Porosity (%)**	**Process Efficiency (%)**
N2, +635 Mesh	0.64	0.12	1
N2, +635 Mesh Heat Treated	0.46	0.10	2.1
He, +635 Mesh	1.48	0.08	3.7
He, +635 Mesh Heat Treated	0.94	0.04	4.7

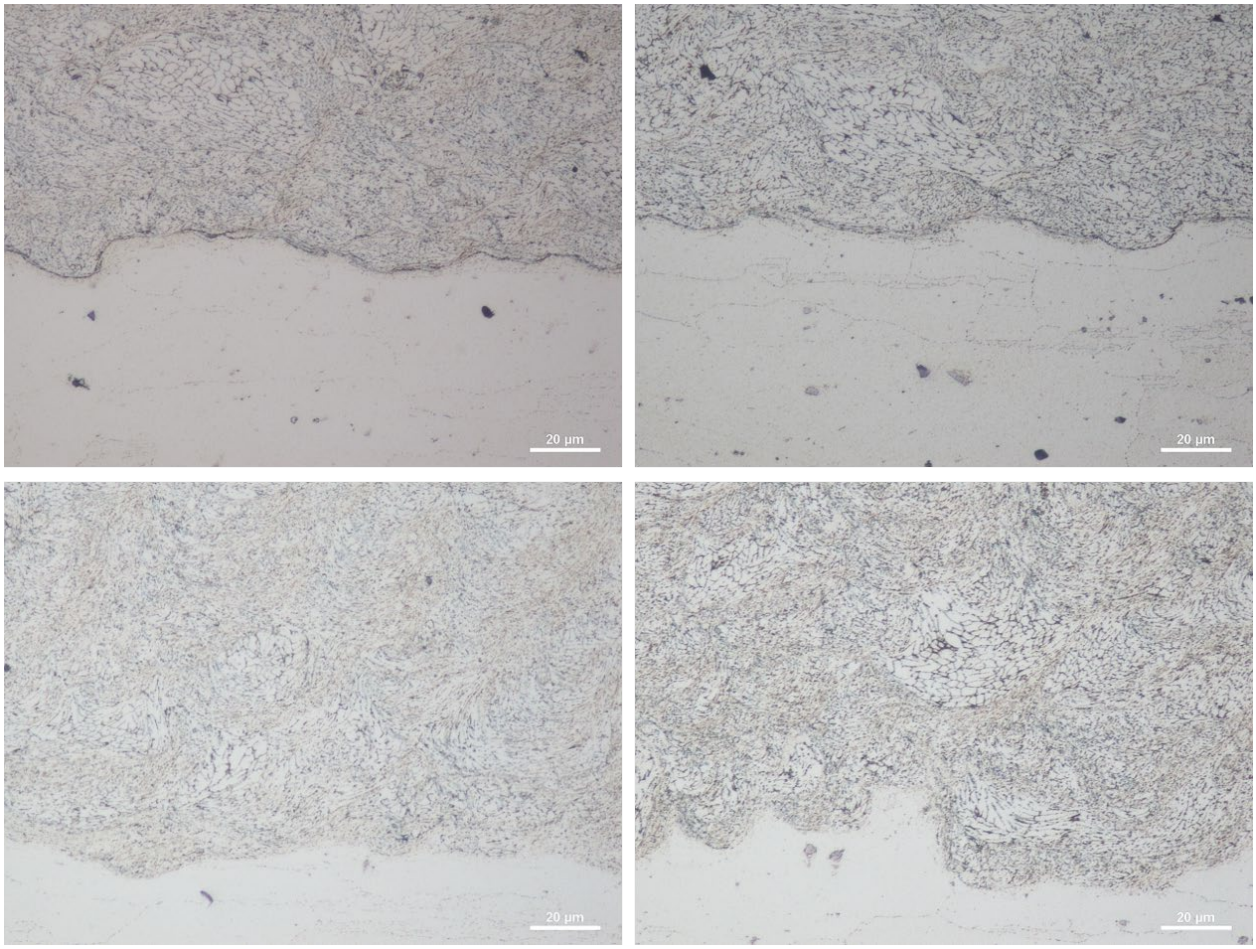


Figure 4. Optical micrographs of etched AA7050 cold spray deposits produced with (top left) as-received powder with N2 gas, (top right) heat treated powder with N2 gas, (bottom left) as-received powder with He gas, (bottom right) heat treated powder with He gas.

3.4 Adhesion

Adhesion tests were performed following ASTM D4541-17. Adhesion tests were performed on size different combinations of powder condition, main process gas and spray nozzle control. Tests are identified by the main process gas (N-nitrogen or He-helium), how the spray nozzle was controlled (R-robot and H–hand-held) and powder condition (S–sieved and T-thermally treated). For example, the spray with He as the main process gas, robot controlled spray nozzle and thermally treated powder is identified as HeRT. The six different adhesion tests were designated as NRT, HeRT, NRS, HeRS, HeHS and NHS. The reported adhesion values are the average of the four samples from each substrate. The failure mode for all samples tested was at the adhesive/dolly interface. Adhesion values, shown in Figure 5, were between 71.9±8 MPa and 83.7±3 MPa. The results show that high adhesion values can be obtained for both the process and unprocessed powders, with He and N2 as the main process gas and using a robot or hand-held spray nozzle. The high adhesion values show that additional testing is need to evaluate the deposition characteristics.

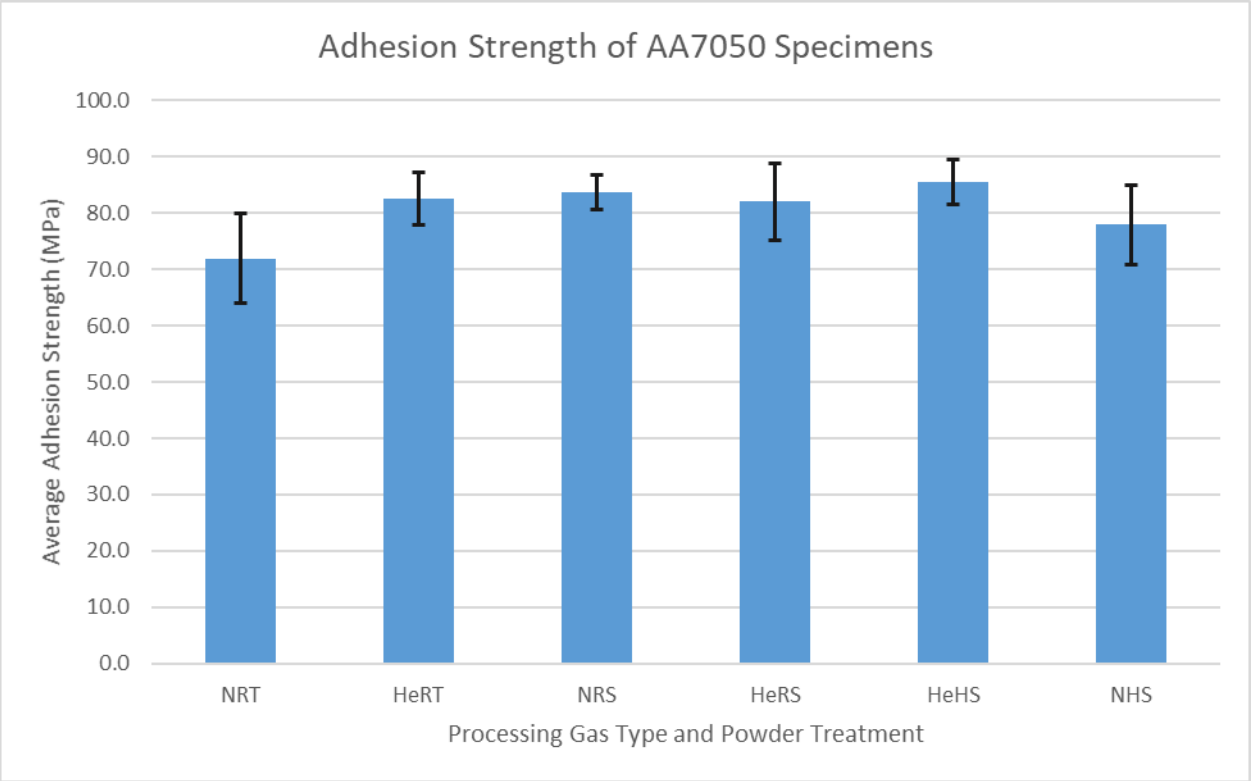


Figure 5. Average adhesion strength for the AA7050 test articles produced using nitrogen and helium as the main process gas and powders in the as-received and in the as-received and thermally treated condition.

3.5 Shear Data

Shear testing is a less expensive and quicker method to evaluate the performance the depositions that tensile testing. Samples were prepared following MIL-J-29445. The same combinations of powder, main process gas and spray nozzle control were used to produce samples for shear testing. The values reported are an average of the two lugs on a single shear samples. Additional testing is needed to get better statistical values for the shear strength. Results of the shear tests were compared to shear strength values reported in the literature and with wrought material tested at the same time the shear samples were tested. The literature reported a shear strength of 303 MPa and the wrought material tested has a shear stress of 270 MPa. The difference is most likely due to the difference in testing methods. The results, plotted in Figure 6, show the shear strength of deposits produced using He for both conditions of powder and spray nozzle control are significantly higher than the shear strength of the deposits produced using N2 (184-225 MPa compared to 71-80 MPa). The shear strengths for the He produce samples are very promising for structural repair and could be improved through process optimization. R Additional work on powder processing and process optimization are needed to improve the shear strength of deposited material produced using N2 as the main process gas. Results from the robot and hand-

held spray nozzle were similar for both N2 and He samples. Stress-strain data were not collected during the testing.

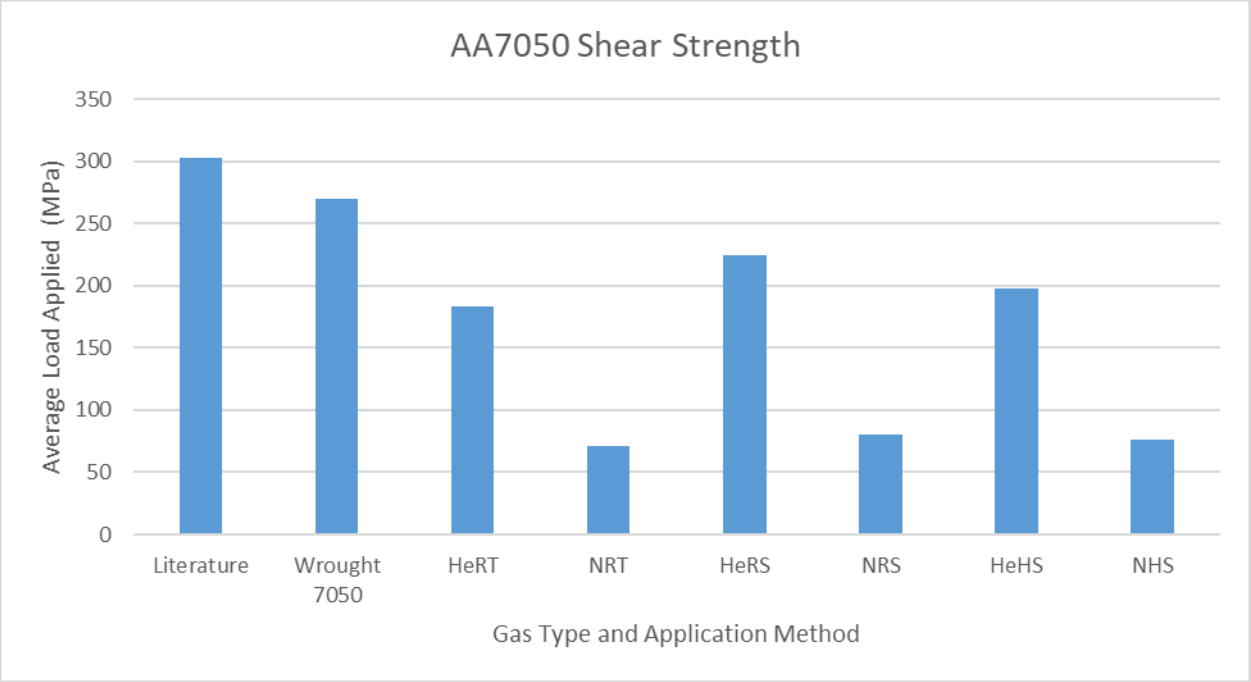


Figure 6. Shear test results for wrought and AA7050 depositions produced using the following: He Robot Thermally Treated Powder (HeRT), N2 Robot Thermally Treated Powder (NRT), He Robot As-Received Powder (HeRS), N2 Robot As-Received Powder (NRS), He Hand-Held As-Received Powder (HeHS) and N2 Hand-Held As-Received Powder (NRS).

4. CONCLUSIONS

The work performed was to evaluate the effects of main process gas, thermal treatment and deposition control on the properties of Cold Sprayed AA7050 powder. The results will be used as the basis for further development of the Cold Spray process for structural repairs using AA7050. The conclusions from this work for deposition of AA7050 on 7050-T7451 are:

- High adhesion strengths (> 70 MPa) can be achieved with both helium and nitrogen as the main process gas.
- Deposition efficiency is higher when using helium compared to nitrogen as the main process gas
- Thermal treatment greatly improves the deposition efficiency for both helium and nitrogen as the main process gas
- SEM images did not reveal an changes in microstructure after thermal treatment
- High shear strengths (> 175MPa) can be achieved with helium gas compared to nitrogen as the main process gas
- Cold Spray process parameters need to be optimized to improve the mechanical properties of the AA7050 depositions

- Fatigue and tensile testing need to be performed as part of the development of a structural repair process
- Depositions produced using helium show excellent promise for structural repair

5. ACKNOWLEDGEMENTS

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