Experimental Analysis and Simulations of the Effects of Deposition Conditions on Deformation Characteristics of Al-6061 Powders in Cold-Spray

A Thesis Presented

by

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To

The Department of Mechanical and Industrial Engineering

In partial fulfillment of the requirements

for the degree of

Master of Science

in the field of

Mechanical Engineering

Northeastern University

Boston, Massachusetts

August 2017
Abstract

Cold spray is an additive manufacturing method based on impacting metal powders on a substrate with the aid of a supersonic gas, and it is used extensively to rebuild a damaged metallic structures. The characteristics of interaction between the coating and the substrate depend on the impact velocity, particle and substrate temperatures, and metallurgical properties of the initial powder. In this work, micron scale Al-6061 particles were sprayed on flat substrates of the same material under different spraying conditions with low particle density to characterize single particle impacts. Three groups of cold sprayed samples with different types of heat treatments were investigated. Three different driving gas conditions were tested: He-gas at 927 m/s and 547 K (He-V927), He-gas at 767 m/s and 400 K (He-V767), and N2-gas at 617 m/s and 582 K (N2-V617). Initial powder and impacted particles were analyzed in intact and cross-sectioned form by oblique Scanning Electron Microscopy and Atomic force microscopy. Tape test, scraping test, and shear test were conducted to collect deformed particles. Fully bonded, partially bonded, and failed particles were systematically observed. Continuum simulations of impact of spherical and non-spherical particles under the same spraying condition of the samples were carried out.

The powder used in the tests has distinct solidification boundaries on the surface with some having large melted segments. Both of features are attributed to the gas atomization process used in powder manufacturing. The surface roughness of the bottom of deformed particles is considerably reduced in all roughness measures, after the impact, in comparison to that of the initial powder. Each test group with different gas conditions show different deformation trends. Splats of He-V927 were flattened and bonded with coronal jets. Splats of He-V767 appeared bonded but the particle remained convex above the substrate, and the splats with N2-V617 had mostly craters on the substrate and some partially bonded particles. The specially heat treated sample group showed mixed jets from both of the particle and the substrate, on the other hand, the other two groups showed mostly substrate jets. The yield stress of the Johnson-Cook model for the substrate was chosen to be 1.75 times that of the particles based on comparisons with experimental results. Simulations allowed further classification of the deformation characteristics as functions of impact velocity and temperature. It showed that with increasing impact velocity deformation progresses from local deformation at the impact interface to lateral spreading of the particle, to substrate jetting and finally to jetting in both sides. This work advances our knowledge in particle deformation in cold spray applications and provides further calibration tools for mechanics models of high-strain rate impacts of metals.
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ACKNOWLEDGMENTS

I provide my sincerest appreciation to Professor Müftü for his support and trust in my research. His trust allowed me to work in innovative ways. He offered me chances to question the research, and find the answer in progressive ways. I am thankful for his efforts to develop my strong aspects, and supplement my weak points through active communication. What I’ve learned from him will be one of the most valued assets in my academic career. I also would like to acknowledge Professor Gouldstone for his expertise. He was always happy to actively discuss the research, and was optimistic about future results and work. He encouraged my work, and allowed me to be independent while steering me in the right direction. Both mentors have been an integral part of the work since its inception, and it was my honor to work with both advisors as a team.

This work was financially supported by the United States Army Research Laboratory through grant number W911NF-15-2-0026. Any opinions in this thesis are those of the author and do not necessarily reflect the viewpoints of the funding agency. The samples used in this work were provided by Mr. Aaron Nardi and Dr. Xuemei Wang of the United Technologies Research Center, CT.

I thank all my colleagues the Applied Mechanics and Tribology Laboratory Qiyong, Enqiang, Ozan, Runyang, TingTing, TJ, and Soroush for a fruitful work environment. I also would like to extend my thanks to Professor Teiichi Ando, Azin Houshmand, and Wendy Evans for their support they have given me during my graduate studies.

I would like to thank Vincent Capone for his extensive help as my tutor and my best friend throughout my life in the US.
I dedicate this thesis to my mom who guides my life, and provides unconditional faith in me. I thank her for all of her support as my mom, mentor, and friend. My best respects, as always.

In addition, thanks to my Amitayus Buddha and all the family of the Bul-Sa Shrine for encouraging me constantly.
Chapter 1 Introduction

1.1 Background

Mechanical systems consist of structural and functional parts. Structural failures can become critical as they impact successful operation of the systems. Major structural failures include distortion, deformation, corrosion, erosion, and fracture due to external loads or residual stresses. Spraying methods have become effective to restore failed structures without repeating the manufacturing process.

Cold Spray (CS) technology has been at the forefront of the spraying technologies due to having many advantages compared to the thermal spraying processes. In CS, the resulting coating retains the properties of the initial particle materials, and induces low residual stresses; the surface preparation requirements are minimal; and the substrate temperature is not elevated significantly [1]. In addition, the cold spray process has higher operational safety due to the absence of high-temperature gas jets or explosive gases, and the cold spray equipment system is simpler than that of thermal spray, as the cold spray process uses solid state particles, not melted materials [1]. With these significant advantages, the cold spray technology finds applications in diverse industrial fields.

Impact of micron scale particles on a surface results in two states: erosion and coating. The goal of the research in to the CS process in general is to find process and parameter combinations that minimize erosion and enable coating. The deposition efficiency, porosity and the bonding quality between particles and the substrate determine the success of the cold spray. An increase in bonded and merged particles with the substrate at the interfaces signifies that the coated structure
operates similarly as the original structure. Numerous studies have investigated well-deposited cold spray splats, but do not focus on subdividing the bonding levels. Conducting research on the bonding levels between erosion and full bonding is a key process to obtain the most effective spraying conditions. Characteristics of partial bonding and morphologies of the deformed particles by impact offer important criteria to evaluate the quality and to control the error rate of the cold spray process.

1.2 Objectives and Problem Description

The transition process from erosion to bonding is the critical step to form the coating in cold spray process. The transition velocity is called the critical velocity, and the characteristics of interaction between particles and the substrate are significantly changed as the particle velocity becomes faster than the critical velocity [1].

This study was motivated by the observations of particular shape patterns of the deformed particles after impact in cold spray. The deformation characteristics of the particles after impact and the level of bonding appear to be related. Major features used to analyze the bonding quality are the contact area, the penetration depth, formation of jets or pileups, surface roughness, and the overall shape of the deformed particles. The advanced cold spraying conditions leading to improve bonding quality were investigated by researching the deformation characteristics of failed particles, partially bonded particles, and fully bonded particles, and using FEA with diverse particle shapes.
1.3 Arrangement of the Thesis

This thesis is composed of six chapters. Chapter 1 presents a concise background on the Cold Spray process, and the motivation and objectives of this research. Chapter 2 provides a literature review related to the study. Chapter 3 introduces the materials used for this research, and describes the experimental methods. The results of the experiments and the significant characteristics of the deformed particles are presented in Chapter 4. The results of a finite element study on the effects of the initial yield strength (the parameter A in the Johnson-Cook model) of the substrate on the deformation are discussed in Chapter 5. Finally, Chapter 6 finishes this thesis by giving a summary presenting the conclusions, and proposing work for future studies. The appendices provide the larger data sets of the experimental results.
Chapter 2 Literature Review

2.1 Trends in Cold Spray Research

Among spray methods, cold spray is a cost effective and quick method to repair and recover mechanical structures [2]. As cold spray is a commercialized technology, academic research aims to improve the quality of bonding and the deposition efficiency.

Depending on spray methods, optimized particle sizes vary [3]. The kinetic spray process, with particles over 50 \( \mu \text{m} \) in diameter and as large as 200 \( \mu \text{m} \) in diameter, exhibits relatively high deposition efficiency. Conversely, the cold spray is limited to particle diameter less than 50 \( \mu \text{m} \). The critical velocities of particles over 50 \( \mu \text{m} \) are significantly slower than the critical velocity of smaller particles.

Major factors which determine the bonding quality of cold spray are gas pressure, gas temperature, standoff distance, and impact velocity [4]. Oxidization is as an important factor for bonding as the particle temperature, and the adhesive strength lowers as a particle becomes oxidized [5]. The bonding occurs when the impact velocity of the particle is higher than the critical velocity of the material and the particle size [6]. The particle mass loading depends on the traverse speed, a critical factor for multi-particle impact [7]. However, the traverse speed for single particle impact is short enough not to consider this as a factor for the bonding quality.

As the curvature along the interface develops, the coating and the substrate are mixed with wave flow by impact [8]. This wave flow affects not just the bonding but also the generation of pileups and jets. One of the key steps of the bonding process is thought to be local melting. The substrate temperature is somewhat raised by impact [9]. The local melting results from the
adiabatic shearing and the thermal effect of the driving gas [10]. The local high stresses and fast
temperature rises influence the local microstructure [11]. The thermal energy is accumulated
during the deformation in the adiabatic state [12]. The localized adiabatic shear instability at the
boundary among particles and the contact surface is thought to cause metallurgical bonding, and
to enhance the coating cohesion [13, 14].

While the bonding quality is the most important factor for cold spray, the deposition
efficiency is also one of the major factors. Recently, cold spray with particles of diverse materials,
including synthetic materials, were investigated to improve the bonding quality of cold sprayed
particles [2]. Coating with the materials as well as coating between different materials was
investigated to improve deposition efficiency and the bonding quality [15]. Successful mixed
material coatings included combinations of ultra-high molecular weight polyethylene and nano-
ceramic composite [16], WC-Co and Ni [17], ceramic titanium dioxide layers on metal surfaces
[18], binary powder composites with Al-Sn [19], Ni and Al composite [20], and aluminum
composite coating reinforced by carbon nanotube [21]. Testing of the multi-material composite
coatings in friction mode showed that the adhesive strength is higher due to the peening effect
[22].

2.2 Relation between Morphology and Velocity

Impact velocity of particles determines the deposition efficiency and the bonding quality.
Sticking happens when the impact velocity nears the critical velocity, but bonding occurs when
the impact velocity surpasses the critical velocity. In cases where the velocity is lower than the
critical velocity and the particle sticks on the substrate, jets come from one of either particles or
substrate [23]. The critical velocity for cold spray varies depending on the particle size. Bigger
particles have lower critical velocities than smaller particles [10]. Moreover, cold-sprayed
titanium coating characteristics were shown to be related to both the impact velocity and the ratio of particle velocity to critical velocity [24].

Increased penetration means broader contact surface area between the particle and the substrate. Penetration depth increased with velocity, and jetting in the particle was suppressed due to much greater absorption of impact energy by the substrate deformation [25]. The result of the strain hardening effect on the contact of a rigid ball and elastic-plastic flat through indentation study showed that the relation between normal load and the penetration depth is linear [26], but the actual contact surface of the ball with the surface is not linear. This represents that particles with higher velocity that have larger impact energy, penetrate deeper and the top of the particle is flatter. It should be noted that although a particle penetrates the substrate deeply, this does not mean that the particle has linearly increased contact area with the substrate.

2.3 Relation between Morphology and Surface Roughness

Cold spray researchers have found that surface roughness correlates to the friction behavior between the impacted particle and the substrate, and to the nano-scaled deformation. According to Sakaki et al., both deposition efficiency of copper and aluminum coatings slightly increase as the surface roughness increases in a certain range (0.2-7 μm Ra) [27]. The surface roughness is lowered after impact as particles crush the peaks nearby the valleys [28]. This is demonstrated by investigating the surface roughness of the bottom of the mushroom shaped deformed particles, as will be discussed later. Yildirim et al. showed that as the particle size and the impact velocity increases, the effect of the surface roughness decreases for particles with diameters of 216, 432, and 864 μm [28]. However, since most of the particles for cold spray are smaller than 50 μm, the effect of the surface roughness is an important factor for plastic deformations. The substrate
surface roughness may simulate metal coating deposition in the gas dynamic spraying process at relatively low accelerating air stagnation pressure [29].

2.4 Relation between the Deformed Morphology and Particle Shape

Metal particles used for cold spray are not single grain. Typically a micro-scale particle is composed of small grains. The size of the grains is about 2.8 μm among 45-90 μm aluminum particles [12]. As the grain size of a particle becomes smaller, its ductility becomes higher [30]. This suggests that the thermal softening by impact could cause better bonding. The softer and coarse-grain conventional powders experience generalized adiabatic shear instability upon impact, resulting in a dense coating, whereas, the harder and fine-grain powder did not exhibit the same extent of deformation, resulted in porous coatings [31, 32].

While the small grain of a particle affects the morphology after impact, the effect of the overall shape of a particle is a significant factor of the morphology. The effect of particle shape has been studied by using different methods. The effects of powder characteristics on coating micro hardness or coating porosity were investigated [24]. Because the powder characteristics affect the impact velocity, and irregular particle presents higher flying velocity than the velocity of spherical particles with the same size, the powder characteristics have significant effects on the morphology after impact [33]. The flattening ratio of the particle after impact depends upon powder morphology [34]. The shape and aspect ratio of a particle, which affect the impact response, are also about the effect of the initial orientation [35]. The effect of powder characteristics can be investigated by the crater shapes, or by indentation with non-spherical indenter [36]. In general, the particle shape is a major factor in coating morphology [37].

As mentioned in Section 2.1, powders for cold spray should fall in a certain size range to improve deposition efficiency. The size effects in impact dynamics are considered by an
optimized size distribution [38]. A smaller particle size distribution presents higher acceleration and higher impact velocity [31], and the smaller aluminum particles lead to better coating because of the peening effect by higher kinetic energy [39]. Finer aluminum powder sizes that yield higher particle velocities also yield higher density deposits, which result in increased ductility compared with coarser powders [40]. Conversely, the bonding quality increases as the particle size is larger under the same impact velocity, as the maximum temperature and the bonding time are both increased.

2.5 Relation between Deformed Particle Morphology and Driving Gas

The effects of the driving gas can be segmented in three categories: type of gas, gas pressure, and gas temperature.

Driving gas should be a cost-effective because it is consumable. Two gases are typically used in cold spray process. Under the same conditions, coating with nitrogen as a driving gas has higher deposition efficiency than the coating with helium [41]. However, as the driving gas affects plastic deformation, mixtures of the two gas are used to improve the bonding quality. Balini et al. studied the effect of driving gases on cold-sprayed aluminum coating, and the results show that He (100 vol.%) processing generated higher hardness values, indicating a compact structure and higher degree of plastic deformation in comparison to processing with addition of 20 vol.% N₂ [42].

The velocity of cold sprayed particles depends not on the gas temperature but on the gas pressure, and the efficiency can be increased by increasing gas pressure and increasing substrate temperature [43].
Driving gas is the only source of direct heating on the particles and the substrate. The driving gas is the only source of the kinetic and the thermal effects including higher impact velocities [44] and improved particle adhesion [45]. The higher driving gas temperature results in higher particle impact temperature [46]. The properties of coating can significantly be improved by using higher driving gas temperatures [38], and the higher gas temperature and pressure increase the deposition efficiency of coatings [27]. At low gas pressure, the surface temperature at the impact point is affected significantly by the driving gas temperature, as the deposition efficiency is influenced by the impact velocity and the surface temperature [47]. As the temperature of splats increases, the flow stress at the interface is reduced which leads to the best inter-splat bonding state [48]. In addition, increasing particle impact temperature decreases the critical velocity of the particle. The substrate temperature influences on the deposition efficiency, bonding and the residual stress [49]. In the present study the substrate was exposed to gas for a short duration and thus can be assumed to be at room temperature. On the other hand, the particles were in the nozzle with the gas and were heated by the driving gas.

2.6 Relation between Morphology and Particle Heat Treatment

The post-heat treatment [50], or post-annealing [51], improve the bonding quality between the particles and the substrate by intermetallic formation. The post-heat treatment of coating surface leads to significant decreasing of coating porosity [52], and the hardening effect is achieved by recrystallizing and resizing grains of cold-sprayed coating [53]. For example, the vacuum heat treatment significantly influenced the microstructure and microhardness of cold spayed coatings [54]. However, post-heat treatment is not always possible depending on the characteristics of the sprayed parts.
The pre-heat treatment before impact obtains the same effects of the post-heat treatment, and the heat treatment temperature significantly influences the bonding characteristics at the interface between the particles and the substrate [55]. The pre-heat treated powders decrease the critical velocity, as the ductility of a particle is increased, and increase the deposition efficiency [33, 56].

Depending on the type of the heat treatment, microhardness decreased as the temperature for heat treatments increased [57]. The hardness of the substrate has a significant effect on the quality of cold spray coating as much as the impact velocity, and more than the spraying distance [58]. Zhang et al. concluded that the most successful initiation of bonding of aluminum particles onto substrates of low roughness by cold spraying requires a metallic surface with a hardness higher than that of the particles [59]. Although the substrate is remarkably hard, coating can be formed with large enough particles in the stream [37].

2.7 Experiments

Single particle experiments have been conducted to measure bonding strength between a splat and the substrate. One experiment investigated the effect of deposition conditions of Ti coatings [9]. By analyzing the force displacement curves when the splat was detached, adhesion strength was calculated. The results show that smaller particles have better adhesion strength. The range of the adhesion strength of particles, with the pre-impact diameter between 10 and 20 μm, was 250-300 MPa; bigger particles with 40-60 μm had adhesion strength ranging between 150-250 MPa. The shear force could be minimized in the analysis, because the resultant graph shows triangles based on the level of friction force of the surface of the substrate.

Various experiments of cold spray samples with multi-particles have been conducted to investigate bonding strength. The most common method to measure adhesion strength is a tensile test. Various investigators performed tensile tests [46, 60-62]. This test used a hydraulic
adhesion/tensile equipment (PAT, DFD Instruments, Kristiansand, Norway) with the circular coated specimen glued to the test element with a heat-cured epoxy resin [61]. The tensile behavior of the material sprayed at the higher temperature revealed strong bonding between particles in the as-deposited material in a direction perpendicular to that of the particle impact vector [62]. Binder et al. conducted the tensile and shear tests [46]. The shear test is similar to the shear test which will be introduced in Chapter 4, which uses a blade for the shear test. Diverse test methods were introduced to measure the adhesion strength: crack test [63], pull-off test of copper particles on aluminum [64], fatigue test with a Wohler rotating vend fatigue testing machine [61]. Each experiment required optimized specimen, for example, E. Sansoucy et al. used triangle-shaped cantilever specimen for the fatigue test, which was grit-blasted and coated one of the sides [65]. The results showed that the substrate was fractured. However, it is possible that the design of the specimen impacted the results.

Besides tensile test, diverse tests have been conducted to investigate the adhesion characteristics. A friction test was conducted to investigate microhardness and tribological behavior of cold-sprayed tin bronze coatings by using turn table [66], and LASAT (Laser Adhesion Test) of cold-sprayed Cu onto Al showed that Cu-Cu inter-lamellar strength was about the same order as that of Cu-Al coating-substrate adhesion [32]. T. Stoltenhoff et al. demonstrated that recovery and recrystallization determine further microstructural developments in cold-sprayed coatings by conducting annealing experiments and respective analysis [67]. Semi-destructive hole-drilling and layer growing curvature methods were applied to measure residual stress [68], and microstructure, thermo-mechanical properties, and residual stress were measured by using neutron diffraction data [69].

Depending on the structure of the substrate, a nozzle cannot access a point with a perpendicular position. The impact angle between the particle and the substrate affects the
deposition efficiency [23]. As spraying perpendicular to the substrate surface, porosity is low and flattening ratio and shear strength gets higher [46]. Moreover, when the nozzle is aimed to be perpendicular to the substrate, the impact angle of each particle is not a major factor of the deposition efficiency. For this reason, only perpendicular particle impact simulations were conducted in the present study.

2.8 FEA

FEA has been used to further investigate the results of experiments by applying the same conditions to specimen. Particle shapes affect the morphology after impact, elliptical Ti particles were used instead of spherical particles to investigate the effect of particle shapes [70]. The results show that irregular particles cause non-trivial deformation patterns due to their random shapes, and the critical velocity of the particles may be higher than that of spherical particles. The material properties are important for the characteristics after impact. Anisotropic properties were applied to assess mechanical characteristics compared to isotropic properties [71]. However, isotropic material properties were applied in this thesis because the samples used for this thesis were made with Al 6061 for the particle and the substrate.

The simulation results of King et al. showed the interface temperature, between copper particles and aluminum substrate, increased when adiabatic shearing reached the melting point, and the fraction increased as the velocities 430 m/s and higher [25]. Melting was confirmed experimentally. This melting is thought to be one of the steps for the bond formation. Although the FE results of the thesis predominantly show bonding at the center of the interface [72], SEM images show that bonding happens more along the sides of the interface. As the study proves, regardless of the substrate material, a thin void was always observed at the center-bottom between a particle and the substrate.
2.9 Mushroom

Later in this work we liken a particular shape of a deformed particle to a mushroom, for the lack of a better terminology. Mushroom formation has not been fully studied, but mushrooms and the conditions that form mushrooms and pileups have been observed in the studies of velocity effects. For example, for copper particles onto the stainless steel substrate at low velocity (about 400 m/s) mushroom shape has been observed [2]. The out-flow of the particle jet appears as a mushroom, and shows evidence of bonding [73]. However, according to SEM images, mushrooms appear when particles deform on the substrate. In addition, according to the tape test results, mushrooms have minor bonding strength. Dykhuizen et al. showed that ledges of the pile-ups are sharper when the particle is bonded [2], but those of empty craters are smoother. Moreover, greater impact velocity results in deeper craters. This is the same result as the samples investigated in this thesis.
Chapter 3 Materials and Methods

Al-6061 powder was sprayed on Al-6061 substrate by using an XYZ system at United Technologies Research Center (UTRC) East Hartford, CT, USA. The size distribution of powder was controlled to a relatively tight range by sieving through 400 mesh. The resulting size distribution is in the range of 40 - 45 μm. The powders were manufactured by gas-atomization technique by Valumet (Stockton, CA, USA) [74].

The Al-6061 powder was also subjected to heat treatment. At the end three different groups of heat treated samples were tested:

- Group-1, CS-16-054: as received no specific heat treatment.
- Group-2, CS-16-055: heat treated for 1.5 hours at 230° C.
- Group-3, CS-16-056: homogenized at 400° C for 6 hours and full solution treated to T4.

These samples were sprayed under three different gas conditions:

- He-V927: Helium gas with average velocity and temperature of 927 m/s and 274° C, respectively.
- He-V767: Helium gas with average velocity and temperature of 767 m/s and 127° C, respectively.
- N2-V617: Nitrogen gas with average velocity and temperature of 617 m/s and 309° C, respectively.

The critical velocity ratio (CVR) is defined as the impact velocity of the particles to the critical bonding velocity. The CVR values for these three different gas conditions were 1.533, 1.072 and 1.075, respectively. The gas conditions are summarized in Table 1.
All of the nine combinations of the three types of powder and three gas conditions were used in the experiments. Care was taken to obtain very sparse coatings on the substrate in order to observe single particle impacts (splats). Cross-sections of the splats were obtained by the members of the Advanced Materials Processing Laboratory (PI, Professor Teiichi Ando) by using a standard slicing, polishing and etching technique for Al-6061. Powders in Group-1 and -2 were obtained from UTRC. The powders and sprayed samples and their cross-sections were imaged in detail by a scanning electron microscope (SUPRA 25 FESEM, Zeiss, Peabody, MA, USA). The morphology of some of the splat samples and the craters on the substrate were also quantified by using an optical microscope (Veeco, Plainview, NY, USA) and an atomic force microscope (Park XE7, Seoul, Republic of Korea).

The splats were subjected to pull-out test by using carbon tape and shear test by using a universal testing machine (UMT Multi-Specimen Test System, Campbell, CA, USA). The steps of the tape test process were as follows:

i. Clean up the sample by using an ultrasonic cleaner (Fisher Scientific FS20D)

ii. Place a piece of carbon tape (EMS, Hatfield, PA, USA) on a targeted zone of the sample (Figure 22a)

iii. Press the targeted zone between pieces of sponge with a vise for 5 minutes (Figure 22b)

iv. Detach the piece of carbon tape quickly by holding one side of the sample

v. Observe the overall targeted zone on the carbon tape by using a magnifying camera (CAPRA OPTICAL, Boston, MA)

vi. Put the carbon tape piece in a SEM to observe the detached splats

vii. Observe the surface roughness of the bottom of the mushroom by using an optical profiler (VEECO)
Shear tests of individual particles were conducted by using the CETR-UMT-2 (Campbell, CA, USA) micro-tribotester. A special indenter was designed in order to apply the appropriate level of normal stress on the substrate. The scraper shown in Figure 1 was made of A6 tool steel and was sharpened to the final shape by hand using a honing stick (Arkansas, USA). In order to determine an appropriate size for the scraper radius it was assumed that it has a cylindrical contour when in makes contact with the substrate Figure 1a. The maximum contact pressure, at the center of contact, can then be determined, for elastic contacts as follows [Mechanical Design (2004), Ansel C. Ugural, USA, New Jersey Institute of Technology, Chapter 3, Table 3.2][Johnson, K.L. (1985), Contact Mechanics, Cambridge, UK, Cambridge University Press][ME EN 7960 – Precision Machine Design Topic 7 tutorial, University of Utah]

\[
P_{\text{max}} = \frac{2F}{\pi b L}, \tag{3.1}
\]

where \( F \) is the applied force, \( L \) is the depth of the cylinder (tool), and \( b \) is the half-width of contact which can be found as follows,

\[
b = \frac{\sqrt{\frac{4F\left[\frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}\right]}{\pi L \left(\frac{1}{R_1} + \frac{1}{R_2}\right)}}}{\pi l(b)} \tag{3.2}
\]

where \( E_1, E_2, \nu_1 \) and \( \nu_2 \) are the elastic moduli and Poison’s ratios of the two materials, respectively, and \( R_1 \) and \( R_2 \) are the radii of contact. Equation (2.1) is used to design the tool by setting the maximum pressure \( P_{\text{max}} \) to the indentation hardness \( H = \sigma_y/0.31 \) of the softer material, which is Al-6061 in this case. Note that \( \sigma_y \) is the tensile yield strength. For a flat substrate \( R_2 \) is set to infinity. Based on this approach, we find the tool radius \( R_1 = 30 \mu m \) for a vertical load of 0.2 N, and tool width of \( L = 200 \mu m \). The tool radius becomes, \( R_1 = 67 \mu m \) for a tool width of \( L = 190 \mu m \), for the decreased load on the substrate.

The scraping/shearing test process involves the following steps:
i. Clean up sample
ii. Set $F_z=0.294 \text{ N, } V_x=2 \mu\text{m/s}$
iii. Place the sample on the table in the UMT machine
iv. Target splats with a magnifying camera
v. Run the process

![Figure 1](attachment:scarper_tip.png)

(a) Hertz Contact between cylinders, (b) Scraper before shear test, (c) Scraper after shear test

**Figure 1** Scarper tip before, during and after the tests. (a) Hertz Contact between cylinders, (b) Scraper before shear test, (c) Scraper after shear test

![Figure 2](attachment:al_coupon.png)

(a) A cold-sprayed Al coupon where carbon tape is applied on the far left group of splats. (b) The carbon tape is gently, and uniformly pressed by using vise-grips.

**Figure 2** a) A cold-sprayed Al coupon where carbon tape is applied on the far left group of splats. b) The carbon tape is gently, and uniformly pressed by using vise-grips.
Table 1. Gas conditions used in the experiments

<table>
<thead>
<tr>
<th></th>
<th>He-V927</th>
<th>He-V767</th>
<th>N2-V617</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas</td>
<td>He</td>
<td>He</td>
<td>N₂</td>
</tr>
<tr>
<td>Average CVR</td>
<td>1.533</td>
<td>1.072</td>
<td>1.075</td>
</tr>
<tr>
<td>Average Velocity(m/s)</td>
<td>927</td>
<td>767</td>
<td>617</td>
</tr>
<tr>
<td>Average Temperature(°C)</td>
<td>274</td>
<td>127</td>
<td>309</td>
</tr>
</tbody>
</table>
Chapter 4 Experimental Results

4.1 Powder Morphology

The SEM images of the group-1 and -2 powders presented in Figure 3 show that powders are not spherical, with some small satellite particles stuck on larger ones. The surfaces of the powders have two general characteristics. Firstly, the solidification boundaries that are typical for gas atomized particles are nearly uniformly distributed. Secondly, some large melt regions are observed on the particles. These regions are also a result of the particle manufacturing process.

![SEM images of a) the powder with the solidification boundaries and b) the powder with the large melt regions](image)

**Figure 3** SEM images of a) the powder with the solidification boundaries and b) the powder with the large melt regions

4.2 Splat Morphology

A great deal of effort went into analyzing and classifying the morphology of the splats and the substrates for all nine combinations of the gas velocities and powder types. Figure 4 - Figure 6
shows the oblique SEM images of the powders in Group-1 through Group-3, respectively, for the three different gas conditions. These figures indicate a strong dependence on impact velocity as determined by the gas conditions. The splats obtained by using He-gas at 927 m/s (He-V927), presented in Figure 4a, Figure 5a and Figure 6a, were flattened and appear to be bonded with a coronal jet region. In contrast, the splats obtained by He-gas at the lower velocity of 767 m/s (He-V767), in Figure 4b, Figure 5b and Figure 6b, appear bonded, but not as much deformed as those deposited by the faster gas. These splats were not buried into the substrate as much and they kept their “curved/spherical” shape above the substrate. The particles delivered by N2-gas (N2-V617) mostly created craters on the substrate and rebounded. A few bonded particles can be observed in Figure 4c, Figure 5c and Figure 6c. These particles are mostly spherical/curved on top of the substrate. At the scale of these images no discernable dependence on powder type can be identified.
Figure 4 SEM images of the splats obtained by using the Group-1 (CS-16-054) powder with gas conditions a) A: He-V927, b) B: He-V767 and c) C: N2-V617.
Figure 5 SEM images of the splats obtained by using the Group-2 (CS-16-055) powder with gas conditions a) A: He-V927, b) B: He-V767 and c) C: N2-V617.

a) CS-16-055A: Flat & Curvature Change at the boundary

b) CS-16-055B: Convex

c) CS-16-055C: Craters
Figure 6 SEM images of the splats obtained by using the Group-3 (CS-16-056) powder with gas conditions a) A: He-V927, b) B: He-V767 and c) C: N2-V617.
Cross-sections of the splats were obtained for a number of particles sprayed with He-V927 and He-V767 for all three powder types. The cross-sections show discretized bonding at the interface with voids. Although particles appeared well bonded to the substrate, voids exist at the center of the interface [75]. It was not possible to obtain cross-sections of the splats deposited with N₂-gas (N2-V617) as they were not bonded strongly. Total of 49 cross-sections were analyzed. The images are provided in Appendix 7. Figure 7 shows a sample of the cross-sections for all three powder types deposited by using He-gas at 767 and 927 m/s. This figure shows particles with faster impact velocity of 927 m/s (He-V927) are flattened on the top, whereas the particles with the slower impact velocity of 767 m/s (He-V767) keep the curved shape. Close inspection of all the cross-sections shows that the impact velocity plays a more significant role in the shape of the deformed particle than the powder type. A summary of these observations is given in Table 2 which confirms this observation.

<table>
<thead>
<tr>
<th>Particle</th>
<th>Impact conditions (m/s, °C)</th>
<th>Total samples</th>
<th>Jets observed</th>
<th>Jets</th>
<th>Particle jets</th>
<th>Substrate jets</th>
<th>Mixed jets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group-1</td>
<td>767, 127</td>
<td>11</td>
<td>10</td>
<td>4/11</td>
<td>1</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>(54)</td>
<td>927, 274</td>
<td>7</td>
<td>2</td>
<td>4/7</td>
<td>–</td>
<td>4</td>
<td>–</td>
</tr>
<tr>
<td>Group-2</td>
<td>767, 127</td>
<td>7</td>
<td>6</td>
<td>5/7</td>
<td>1</td>
<td>4</td>
<td>1</td>
</tr>
<tr>
<td>(55)</td>
<td>927, 274</td>
<td>7</td>
<td>–</td>
<td>5/7</td>
<td>–</td>
<td>5</td>
<td>–</td>
</tr>
<tr>
<td>Group-3</td>
<td>767, 127</td>
<td>8</td>
<td>5</td>
<td>3/8</td>
<td>–</td>
<td>–</td>
<td>3</td>
</tr>
<tr>
<td>(56)</td>
<td>927, 274</td>
<td>9</td>
<td>2</td>
<td>5/9</td>
<td>–</td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>

Table 2 Summary of the observations of cross-sectional images presented in Appendix 6. Particles that impact the surface with 927 m/s are flattened and those that hit the surface with 767 m/s keep their curved shape on the top. Impact of particles from groups-1 and -2 tend to cause jetting in the substrate whereas particles from Group-3 tend to cause mixed jets.
Specific details of the coronal jets in Figure 4–Figure 6 are observed in the cross-sectional images. In fact, not all cross sectional images show distinct jets. In the 49 cross-sectional images shown in Appendix 7, jets can be identified in 26 cases. As these SEM images represent relatively random cross-sectional planes, it is hard to state that the remaining 23 cases did not form jets. Nevertheless, based on these images, three distinct jetting behaviors are identified: the jet that forms only in the particle; the jet that forms only in the substrate; and, a merged jet that originates from both the particle and the substrate. Figure 8–Figure 10 show these jet types for the three powders in Groups-1, -2 and -3, respectively. It is interesting to note that majority of the jets for powders in Group-1 and Group-2 is substrate-jets, whereas for Group-3 the majority is of the mixed-jet type. This indicates that it was easier for the powder in Group-3 to be bonded with the substrate around the periphery of contact, than the other two powder groups.
Figure 7 Typical SEM images of the cross-sections of the splats for the Group-1 (a, b), Group-2 (c, d) and Group-3 (e, f) powders sprayed with He-gas with velocity 927 (He-V927) and 767 m/s (He-V767).
Figure 8 Jetting types observed in the splats for the Group-1 (CS-16-054) powder, with gas conditions He-V927 and He-V767. Empty spot indicates that the particular classification group was not observed.
Figure 9 Jetting types observed in the splats for the Group-2 (CS-16-055) powder, with gas conditions He-V927 and He-V767. Empty spot indicates that the particular classification group was not observed.
Another interesting characteristic of the splats can be observed in the relatively low impact velocity conditions provided by He-V767. Particles that are sprayed under such conditions tend to retain their curved shape on the top and form jets that extend beyond the contact footprint. The resulting particle shape resembles a mushroom. Impact energy needs to be used to deform the particle more than penetrating the substrate to form mushrooms. Bonded mushroom shaped particles are only observed in splats that are deposited the Helium gas at 767 m/s impact speed (He-V767) as shown in Figure 11.
Mushroom shaped, bonded splats are observed in all powder groups sprayed with He-V767. Mushroom shaped particles are also observed in the splats obtained by N2-gas at lower velocity of 617 m/s. These particles are not bonded well to the substrate, and they are the only group that is removable by the carbon-tape test. The adhesion strength of the mushroom is less than that of the carbon tape which is 1,000gf/25mm-width. Figure 12 shows two such splats. In particular, the top view of the splat before removal, the bottom view of the splat after removal and the crater in the substrate are shown. More SEM images of this group of tests are presented in Appendix 1.

Mushrooms deform more on the top of the substrate at the position where pile-ups are relatively smaller around the periphery of the crater. It is interesting to note that the surface roughness in the contact region, i.e. the bottom of the particle and inside the crater is considerably reduced as compared to the original roughness of the particle.
Mushroom shaped splats can be removed from the substrate with a carbon-tape if they are deposited by using N2-gas with 617 m/s (N2-V617).

Surface roughness of the powder and the splats are further examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Representative SEM images of the powder, in Figure 3, show that at the micron (or particle length) scale the predominant surface-features are dominated by the solidification boundaries. In addition, at this scale, some particles have a smoother cover that forms due to rapid solidification of metal during gas atomization process. At the sub-micron scale the powder, the splats and the bottom of the mushrooms were scanned by using AFM. The scan area varies between 1×1 μm to 0.1×0.1 μm. Appendix 8 shows the three dimensional images of the scanned areas. Table 3 shows the roughness measures peak-to-valley, $R_{\text{pv}}$, standard deviation $R_{\sigma}$, average roughness $R_{\alpha}$, and average peak height $R_z$ for the powder, splat and the bottom of the mushrooms. These measurements show that impact causes significant flattening of the surface asperities. The negative values of $S_k$ explains that the
dominant features of the mushroom bottom is valleys, conversely, that of particle is hills. In order to improve the statistical reliability of this conclusion the number of samples in each category should be increased. Moreover, a standard scan size should be used in all the scans. These shortcomings notwithstanding the results presented in Table 3 are exciting evidence of change of surface roughness due to impact.

<table>
<thead>
<tr>
<th>Size (um)</th>
<th>Rpv (nm)</th>
<th>Rq (nm)</th>
<th>Ra (nm)</th>
<th>Rz (nm)</th>
<th>Sk</th>
<th>Ku</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>0.3x0.3</td>
<td>177.00</td>
<td>23.00</td>
<td>17.00</td>
<td>171.00</td>
<td>0.72</td>
</tr>
<tr>
<td>Powder</td>
<td>0.5x0.5</td>
<td>111.00</td>
<td>14.00</td>
<td>10.00</td>
<td>109.00</td>
<td>0.04</td>
</tr>
<tr>
<td>Splat Top CS-16-054 A</td>
<td>0.1x0.1</td>
<td>123.00</td>
<td>20.00</td>
<td>17.00</td>
<td>119.00</td>
<td>0.59</td>
</tr>
<tr>
<td>Mushroom CS-16-055 C</td>
<td>1x1</td>
<td>91.15</td>
<td>10.58</td>
<td>7.80</td>
<td>87.34</td>
<td>-0.35</td>
</tr>
<tr>
<td>Mushroom CS-16-056 C</td>
<td>1x1</td>
<td>82.81</td>
<td>11.77</td>
<td>9.22</td>
<td>79.88</td>
<td>-0.16</td>
</tr>
</tbody>
</table>

Table 3 Surface roughness measurements of the powder, splat and the bottom of the mushrooms.

Oblique SEM images of some of the craters left on the substrate after the removal of the loosely attached particles by carbon-tape are given in Figure 13. As it can be expected, the craters are not exactly spherical due to the imperfections in the particle shape. Moreover, the material pile-up along the edges of the craters is not uniform, showing discontinuity around the periphery. This is attributed to the shape of the particle, and the anisotropic plasticity of the substrate. Note that the pile-ups, which are observed here, were obtained with relatively low speed impacts (N2-V617). Under this condition no jetting in the substrate is observed, conversely, two to four randomly positioned pile-ups exist around the periphery of the crater. As mentioned before at higher impact velocities where the bonding is stronger, the substrate pile-ups develop into jets that potentially merge with the particle jets.
Figure 13 Craters on the substrate following removal of loosely attached particles by carbo-black tape. a) Group-1, b) Group-2, and c) Group-3 N2-V617

4.3 Shear Test Results

Shear test aimed to explore the adhesion strength by scraping splats to break the interface between the splat and the substrate. The cold-sprayed titanium particles of Goldbaum et al. result in hemispherical particles that protrude from the substrate surface. This offers sufficient area to engage the scraper tip. Moreover, the relatively lower contact area between the Ti-particles and the substrate possibly indicates lower bonding force [9]. In contrast, the well-bonded aluminum splats in this work are buried into the substrate. Nearly all attempts to remove the particles by scraping tests resulted in shearing of the particles.

The shear stress was measured as shearing happened on the splats. The shear strength of Al 6061 is 207 MPa. The results of the shear test shows that the shear stresses of the splats were lower than that of Al 6061; the shear stresses varied between 24 to 134 MPa. The friction force was removed from the resultant shear force.
4.4 Scraping Test Results

The goal of the scraping test is to detach the mushroom-shaped splats from the substrates, and to evaluate the overall characteristics.

The resultant graphs from scraping tests of CS-16-055C do not show any perceptible changes in the load-displacement curves. All splats were detached by the scraping test, meaning that the splats on the sample were not tightly bonded. Although the tool pushed splats, detached splats do not show recognizable deformation. Figure 15 shows CS-16-055C Splat #24:

Figure 15 Detached mushrooms by scraping test
The results of the scraping test present that the particle impact conditions of N2-V617 lacked enough energy to penetrate deeply the substrate. This caused the particles to deform on the substrate, as they did not sufficiently penetrate the substrate for bonding.

**Figure 16** The resultant graph of the scraping test with Group #2(CS-16-055C)
Chapter 5 Finite Element Simulations of Experiments

Single particle impacts were simulated with the finite element method by using the commercially available software package ABAQUS/Explicit 6.13-2. The effects of particle impact velocity, material properties, particle’s morphology and process temperature were simulated. The results were compared with the characteristics of the samples in Groups #1, #2 and #3. Morphologies of the deformed particles after impact including mushrooms, craters, jets and pileups were observed.

5.1 FEM Model

5.1.1 Contact and Cohesive Properties of the Interface

The numerical analysis was conducted by creating and meshing the full models of the particles by using the Abaqus graphical user interface. A Python code was used to create the substrate and run the simulations [76].

The effects of particle shape and impact orientation of non-spherical particles were investigated by using numerical simulations. Three different shapes, shown in Figure 21, were considered in the FE simulations. These include a 29 um diameter sphere, a pill-shaped and a peanut shaped particle. The volumes of each of these particles were 12746 μm$^3$, 13068 μm$^3$ and 20405 μm$^3$, respectively. The impact velocity of the spherical particle was normal to the substrate. The long axis of the pill-shaped particle was oriented at 0° and 43° degrees with respect to the surface of the substrate. The long axis of the peanut-shaped particle was parallel to the substrate.
The substrate dimensions were large enough to prevent reflected waves to reach the particle-substrate interface before “rebound.”

Contact and cohesion in the particle-substrate interface were considered. A new VUINTERACTION subroutine specifically tailored for the high velocity impacts prepared by Dr. Lin was used in our simulations [77]. This user subroutine divides the interaction between the particle and the substrate into a compressive and a cohesive state much like the native Abaqus code, but it has the capability to adjust the contact and cohesion parameters more effectively. The contact and cohesion parameters are the contact compression modulus \( k_{com} \), the elastic tensile modulus in cohesion \( k_{coh} \), the cohesion energy \( G \) and maximum traction stress \( \sigma_0 \), as shown graphically in Figure 17.

Figure 17 Graphical representation of the compressive and the cohesive states.

5.1.2 Meshing

Abaqus/Explicit ver. 6.13-2 was used for the impact simulations. Both the substrate and the particles were meshed with the 8-node, 3D continuum (C3D8RT) elements with single integration points for hourglass control. The material was assumed to be isotropically elastic and
isotropically plastic with isotropic hardening. Care was taken in meshing, to ensure that sides of the elements were as close to being mutually orthogonal as possible. Particles were meshed with the same element size \((d_p/25)\). The substrate was discretized with a dense mesh near the contact region and with a coarse mush away. The two regions were connected by using mesh tie constraints, but in order to reduce the influence of stress waves on the bonding between the particle and the substrate only one tie surface was used. The element mesh transition technique to connect the coarse and fine mesh regions helped reduce the total element number of elements and thus the computational effort. Figure 18 shows the particle and the substrate meshes described above. The substrate was assumed to be fixed on the XYZ boundary.

![a) Particle mesh, and b) Substrate mesh](image)

*Figure 18* a) Particle mesh, and b) Substrate mesh

Every case of the model had different impact velocities and temperatures of the particle; however, the temperature of the substrate was fixed at room temperature.
5.1.3 Material Model

Impacts of Al 6061 particles on a flat substrate made of the same material were simulated. The thermal conductivity, elastic modulus, thermal expansion coefficient, and specific heat were considered to be temperature dependent as reported by Chen et al. [78].

Heat is generated through impact process due to friction and plastic deformation of the particle and the substrate. The generated heat is assumed to be equally distributed into the both of the particle and the substrate. As the amount of heat conversion from the plastic strain energy assumed 90% of the energy, the inelastic heat fraction \( \beta \) was 0.9, which works as a heat source of the simulation. However, it is necessary to account for the progressive damage and failure for the material by the large deformation, and element deletion was considered [79, 80]. The damage initiation criteria is reached when the damage parameter \( \omega = \sum \frac{\Delta \varepsilon_p}{\varepsilon_f} = 1 \) [81], where \( \Delta \varepsilon_p \) is an increment of the equivalent plastic strain, and \( \varepsilon_f \) is the failure shear strain. The damage parameter increases as the material is plastically deformed, and the load bearing ability of the material drastically decreases after the damage initiation.

The effects of strain hardening, strain rate hardening and temperature softening on the yield stress of the material were considered by using the bilinear Johnson-Cook model [78].

\[
\sigma_Y = (A + B \varepsilon_p^n) \left( 1 + C \ln \frac{\dot{\varepsilon}_p}{\dot{\varepsilon}_0} \right) \left( 1 - \frac{T - T_R}{T_m - T_R} \right)^m, \quad \text{with} \quad C = \begin{cases} C_1 \text{ and } \dot{\varepsilon}_0 = 1, \text{ if } \varepsilon_p < \dot{\varepsilon}_c \\ C_2 \text{ and } \dot{\varepsilon}_0 = \dot{\varepsilon}_c, \text{ if } \varepsilon_p > \dot{\varepsilon}_c \end{cases} \tag{5.1}
\]

where the parameters \( A, B, \) and \( n \) are the strain-hardening parameters, \( C \) controls the strain rate hardening, \( \dot{\varepsilon}_0 \) is the reference strain rate, \( T_R \) is a reference temperature, \( T_m \) is the melting temperature of the material, and \( m \) is the temperature exponent [82]. Recently, Xie et al. [82] and Alizadeh [76] reported the Bilinear Johnson-Cook model constants as reported in Table 4. These values were obtained by using Al-6061 particles impacting sapphire surface, and thus strongly
reflect the particle properties of the Group-#3 (CS-16-056). Xie et al. report a substantial difference in the grain size of the particle and substrate materials, which is expected to have an effect on the relative hardness. At the present time the JC-properties for the type of Al-6061 used as the substrate are not available. We hypothesize that the grain size difference is most likely to affect the slow-strain yield stress parameter $A$. In this work, we varied only the $A$ parameter for the substrate in the range of $A$ and $2A$ and kept the other JC parameters the same as those determined for the particles.

<table>
<thead>
<tr>
<th>$A$, MPa</th>
<th>$B$, MPa</th>
<th>$n$</th>
<th>$C_1$</th>
<th>$C_1$</th>
<th>$m$</th>
<th>$T_m$, K</th>
<th>$\dot{\varepsilon}_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>270</td>
<td>154.3</td>
<td>0.239</td>
<td>0.002</td>
<td>0.029</td>
<td>1.42</td>
<td>925</td>
<td>597.2</td>
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</tbody>
</table>

**Table 4** Bilinear Johnson-Cook model constants

<table>
<thead>
<tr>
<th>$E_r$</th>
<th>Elastic modulus, GPa</th>
<th>Temperature dependent(<a href="#">Figure 19</a>)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$</td>
<td>Mass density, Kg/m$^3$</td>
<td>2700</td>
</tr>
<tr>
<td>$\nu_r$</td>
<td>Poisson’s ratio</td>
<td>0.33</td>
</tr>
<tr>
<td>$\alpha_r$</td>
<td>Thermal expansion ratio, K$^{-1}$</td>
<td>Temperature dependent(<a href="#">Figure 19</a>)</td>
</tr>
<tr>
<td>$k_r$</td>
<td>Thermal conductivity, W/m·K</td>
<td>Temperature dependent(<a href="#">Figure 19</a>)</td>
</tr>
<tr>
<td>$c_r$</td>
<td>Specific heat, J/kg·K</td>
<td>Temperature dependent(<a href="#">Figure 19</a>)</td>
</tr>
<tr>
<td>$\beta$</td>
<td>Inelastic heat fraction</td>
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<tr>
<td>$T_m$</td>
<td>Melting temperature, K</td>
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</tr>
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<td>$T_R$</td>
<td>Reference temperature, K</td>
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<tr>
<td>$\varepsilon_f$</td>
<td>Failure shear strain</td>
<td>3</td>
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<tr>
<td>$\mu$</td>
<td>Kinetic friction coefficient</td>
<td>0.7</td>
</tr>
<tr>
<td>$\sigma_0$</td>
<td>Initial damage stress</td>
<td>325MPa</td>
</tr>
<tr>
<td>$G$</td>
<td>Cohesion energy</td>
<td>1.0</td>
</tr>
<tr>
<td>$k_{com}$</td>
<td>Contact compression modulus</td>
<td>7.78x10$^{11}$ - 1.52x10$^{12}$MPa/mm</td>
</tr>
<tr>
<td>$k_{coh}$</td>
<td>Elastic tensile modulus in cohesion</td>
<td>1.0x10$^{11}$MPa/mm</td>
</tr>
</tbody>
</table>

**Table 5** Simulation properties
5.2 Results and Discussion

*Effects of Substrate Hardness*

The substrate yield strength was varied in the range of $A$ to $2A$ with increments of $A/4$. Simulations of impact of 29 um diameter spherical particles were carried out for the impact conditions used in the experiments. The experimental and simulations results were compared visually. The jetting, pile up and the shape of the deformed particle were used as the matching criteria. All simulation results are reported in Appendix 10. The best match was observed when the yield strength of the substrate is equal to $1.75A$ or 472.5 MPa, as shown in Figure 20. This value was used in the following simulations.
The effects of particle shape on the deformation and bonding characteristics of the system are simulated by using the shapes and impact orientations described in Figure 21. The simulated impact velocities and temperatures coincide with the experimental conditions. The cross-sections of the deformed particles for the three different gas conditions are presented in Figure 22, Figure 23, Figure 24. Spherical impacts are presented in this group to provide a reference. The results show that the overall characteristics of the deformation are similar to the experimental conditions represented by CS-16-056, under the identical temperature and velocity conditions. The positions
of the jets, pileups or mushrooms, and the bonding positions and the degree of bonding differ depending on the particle shapes, as shown in Figure 22, Figure 23, Figure 24. The results using the peanut-shaped particle showed that the particle was tilted toward the direction of the bigger mass after impact. This caused partial bonding at the interface between the particle and the substrate.

---

![Figure 21](image1.png)

**Figure 21** Particles before Impact, a) sphere(d=29 μm), b) 43° degrees tilted Irregular particle(26x30 μm) with respect to the surface of the substrate, c) 0° degrees tilted Irregular particle(26x30 μm) with respect to the surface of the substrate (26x30 μm) d) peanut-shape particle(d₁=24 and d₂=30 μm)
Figure 22 A: 547K, 924m/s, Flat Top, Mixed Jets

Figure 23 B: 400K, 767m/s, Convex Top, Mixed Jets
Effects of impact velocity (50-1000 m/s) and temperature (100-350°C) on the shape of the deformed particles and bonding were investigated by using spherical particles. All of the results are presented in Appendix A11-1, A11-2 and A11-3. With increasing impact velocity, the shape of the deformed particle transitions to three or four distinct patterns. The velocities at which the transitions occur depend on the initial particle temperature. Along the way, some particle and temperature combinations result in bonding. Following is a brief summary for the 373 K impact temperature. “M” indicates the different morphological states of the system

- M1 occurs in the velocity range of 50 – 150 m/s: The impact interface remains nearly flat, but the top of the particle keeps its spherical shape.
- M2 occurs in the velocity range of 150 – 600 m/s: The substrate starts to show some appreciable deformation but most of the deformation is in the particle. In this range the particle starts to deform more laterally and takes on the morphology of a mushroom at the higher end of the regime. The first occurrence of bonding is seen at 550 m/s.
M3 occurs in the velocity range of 650 – 750 m/s: The depth of the substrate contact area further increases, and its edges form a distinct jetting pattern, while the particle starts to spread over the substrate jet.

M4 occurs in the velocity range of 800 – 1000 m/s: The substrate jetting continues but the particle also develops a distinct jet. It should be noted that at 1000 m/s the top of the deformed particle becomes nearly flat.

The velocities at which the four distinct morphological changes transition from one to another, and the occurrence of bonding has a temperature dependence. This is summarized in Table 6. The laterally spreading shape, also called mushroom, starts at the same velocity irrespective of the impact temperature of the particle. On the other hand, we see that the bonding velocity decreases as the particle temperature increased. The particle jet starts at a lower velocity as the particle jet temperature is higher, conversely, the substrate jet starts at the same temperature as the substrate temperature is constant. Although not shown in this table, but as it can be observed in Appendix 11-1, 11-2, and 11-3, the velocity at which the top of the deformed particle flattens is decreased as the particle temperature increased.

<table>
<thead>
<tr>
<th>Particle temperature at impact (K)</th>
<th>373</th>
<th>423</th>
<th>473</th>
<th>523</th>
<th>573</th>
<th>623</th>
</tr>
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<tr>
<td>M1 50 – 150</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
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<td>50</td>
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<td>M2 150 – 600</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>M3 650 – 750</td>
<td>650</td>
<td>650</td>
<td>600</td>
<td>600</td>
<td>600</td>
<td>600</td>
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<tr>
<td>M4 800 – 1,000</td>
<td>850</td>
<td>850</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td>700</td>
</tr>
<tr>
<td>$V_{cr}$ m/s</td>
<td>550</td>
<td>550</td>
<td>450</td>
<td>450</td>
<td>400</td>
<td>350</td>
</tr>
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</table>

**Table 6** Velocity ranges for the four distinct morphological shapes, reported in m/s. The critical velocity $V_{cr}$ indicates the first occurrence of bonding.
Chapter 6 Summary, Conclusion and Recommendation for Future Work

6.1 Summary and Conclusion

Impact of micron scale Al-6061 particles with a flat substrate of the same material was investigated experimentally and through continuum simulations. Experiments involved three metallurgically distinct particles sprayed under three different spraying conditions. Thus, a total of nine tests representing material properties, impact velocity and temperature conditions were carried out. Sprays were carried out under low particle density conditions to enable characterization of single particle impacts. Impacted particles were analyzed in intact form by oblique SEM imaging, as well as cross-sectional SEM imaging.

External morphology of non-impacted, powder particles were investigated by oblique SEM and AFM imaging. The SEM imaging shows powders are not spherical, and often have smaller satellite particles stuck on. The particle surfaces have distinctly visible solidification boundaries. Some particle surfaces have large melted regions. Both solidification boundaries and the melted regions are attributed to the gas atomization process used in particle manufacturing. The AFM imaging shows that powder particles have at least two levels of roughness that are possibly uncorrelated. The smallest roughness level has Ra = 10 nm and Rq = 14 nm before impact. Later analysis showed that the roughness of the impacted surface of the particle is reduced to Ra = 7.8 nm and Rq = 10.58 nm.

General trends that depend on impact velocity as determined by the gas conditions were observed. The splats obtained by using He-gas at 927 m/s (He-V927), were flattened and bonded with a coronal jet region. The splats obtained by He-gas at 767 m/s (He-V767) appear bonded, but kept their “curved/spherical” shape above the substrate. The particles delivered by N₂-gas
mostly created craters on the substrate and rebounded. Based on cross-sectional images, three distinct jetting behaviors are identified: the jet that forms only in the particle; the jet that forms only in the substrate; and, a merged jet that originates from both the particle and the substrate. It is interesting to note that the majority of the jets for powders in Group-1 and Group-2 are *substrate-jets*, whereas for Group-3 the majority is of the *merged-jet* type.

Attempts to remove individual particles from the substrate and measure the detachment energy failed. Splat deposited by He-gas at 927 m/s (He-V927), and 767 m/s (He-V767), were all sheared-off during these attempts. Shear stress values were not reliable and varied between 24 and 134 MPa. The splats deposited by N₂-gas at 617 m/s (N₂-V617) were for the most part easy to remove. Either application of carbon-black tape, or the scrapers (with unmeasurable levels of force) were able to remove these splats. These impact surfaces of the splats as well as the craters were then imaged. This revealed that the native surface roughness of the splats is considerably reduced after impact, as mentioned above.

Continuum simulations of impact of spherical and non-spherical particles with the impact gas conditions used in the experiments were carried out. The initial yield stress ($\sigma$) of the substrate was chosen to be 1.75 times that of the particles based on comparison of the simulation and experimental results. All features observed in the experiments were reproduced in the simulations, giving more confidence model parameters. By using continuum simulations, four distinct deformation regimes that primarily depend on the impact velocity were identified. These regimes primarily dominated by: i) vertical compression, ii) flow dominated; iii) jetting in the substrate, and iv) jetting in the particle.
6.2 Future Works

This work relied extensively on observations of the deformed morphology of the cold sprayed samples after impact. The relevant impact conditions for forming of mushrooms, substrate and particle jets and bonding were identified experimentally and studied systematically by conducting the finite element analysis. This work can be expanded as follows:

1. Cross-sectional SEM images show that splats are partially bonded at the interface. Bonding is observed mostly at the slope side of the interface, whereas the center of the contact region is typically unbonded. On the contrary, the simulation result showed evenly distributed bonding interface. The mechanism of bonding should be investigated in more detail.

2. The powder used in this study has irregular shapes in four different ways, but the simulated particles are ideally smooth. We showed that the surface roughness changes substantially after impact and could have an important contribution to impact mechanics and bonding. The effect of surface roughness should be investigated theoretically.

3. The experimental methods in this study worked significantly to investigate the morphology of the particles before and after impact, and to verify the simulation results. Although using carbon tape and collecting in isopropyl are good methods to keep particles, it is impossible to freely control the micron-sized particles. Improved particle-collecting techniques and 3D visualization can help better understand the experimental data e.g. mushroom angles, penetration depth.
Appendix 1: Tape Test

Mushroom-Shaped Splat Top / Mushroom-Shaped Splat Bottom / Resulting Crater

- CS-16-054, N2-617 #5

- CS-16-054, N2-617 #8

- CS-16-056, N2-617 #3

- CS-16-056, N2-617 #11
• CS-16-056, N2-617 #14

• CS-16-056, N2-617 #15

• CS-16-056, N2-617 #17

• CS-16-056, N2-617 #21
Appendix 2: Scraping Test

Mushroom-Shaped Splat Top / Resulting Crater

- CS-16-055, N2-617 #4

- CS-16-055, N2-617 #5

- CS-16-055, N2-617 #7

- CS-16-055, N2-617 #8
• CS-16-055, N2-617 #10

• CS-16-055, N2-617 #11

• CS-16-055, N2-617 #12

• CS-16-055, N2-617 #15

• CS-16-055, N2-617 #22
- CS-16-055, N2-617 #23
  ![Image](image1.png)

- CS-16-055, N2-617 #24
  
  After the scraping test, the resulting detached splat was collected.
  
  (Mushroom-Shaped Splat Top / Mushroom-Shaped Splat Bottom / Resulting Crater)
  
  ![Image](image2.png)

- CS-16-055, N2-617 #25
  ![Image](image3.png)
Appendix 3: Cross-Section of Mushrooms

- CS-16-054, He-V767

- CS-16-055, He-V767

- CS-16-056, He-V767
Appendix 4: Veeco– Surface Roughness

Surface roughness of mushroom-shaped splats’ bottom & that of side of the buried part

- CS-16-054, N2-617 #1

- CS-16-054, N2-617 #2
CS-16-054, N2-617 #3

Title:

X Profile

Y Profile

<table>
<thead>
<tr>
<th>N</th>
<th>X</th>
<th>Y</th>
<th>Hb</th>
<th>Unit</th>
<th>Angle</th>
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<tr>
<td>1</td>
<td>0.31</td>
<td>24.00</td>
<td>4410</td>
<td>in</td>
<td>-</td>
</tr>
</tbody>
</table>

Legend:

- Eq: 1.00 mm
- Ee: 1.24 mm
- Ei: 23.56 mm
- Ey: 2.19 mm
- Ei: -5.68 mm

Angle: -
Curve: -41.11 mm
Tight: None
Area: 274.70 mm²
Appendix 5: Veeco– Surface Roughness

Surface roughness of craters / Slope of craters

- CS-16-054, N2-617 #1
- CS-16-054, N2-617 #2
CS-16-054, N2-617 #3
CS-16-054, N2-617 #4
• CS-16-054, N2-617 #5
CS-16-054, N2-617 #6
CS-16-055, N2-617 #1
CS-16-055, N2-617 #2
CS-16-056, N2-617 #1
CS-16-056, N2-617 #2
CS-16-056, N2-617 #3
CS-16-056, N2-617 #4
CS-16-056, N2-617 #5

[Graphs and tables with data points and measurements]
CS-16-056, N2-617 #6
• CS-16-056, N2-617 #7
- CS-16-056, N2-617 #8
CS-16-056, N2-617 #9
Appendix 6: Dominant Splats Type

Dominant Splats Type of Samples

- CS-16-054, He-V927: Flat & Curvature Change at the boundary

- CS-16-054, He-V767: Convex

- CS-16-054, N2-617: Concave
- CS-16-055, He-V927

- CS-16-055, He-V767

- CS-16-055, N2-617
• CS-16-056, He-V927 :

• CS-16-056, He-V767 :

• CS-16-056, N2-617 :
Appendix 7: Cross-Section Images

Cross-Section Image / Jet Origin

- CS-16-054, He-V927 #1: Particle

- CS-16-054, He-V927 #2: Substrate

- CS-16-054, He-V927 #3: N/A
- CS-16-054, He-V927 #4 : Particle

- CS-16-054, He-V927 #5 : Substrate

- CS-16-054, He-V927 #6 : Substrate

- CS-16-054, He-V927 #7 : N/A
- CS-16-054, He-V767 #1 : Substrate

- CS-16-054, He-V767 #2 : N/A

- CS-16-054, He-V767 #3 : N/A

- CS-16-054, He-V767 #4 : Particle
• CS-16-054, He-V767 #5 : Particle (Horizontal Jet)

• CS-16-054, He-V767 #6 : N/A

• CS-16-054, He-V767 #7 : Substrate

• CS-16-054, He-V767 #8 : N/A
- CS-16-054, He-V767 #9 : N/A

- CS-16-054, He-V767 #10 : Substrate

- CS-16-054, He-V767 #11 : Particle

- CS-16-054, He-V767 #12 : N/A
- CS-16-055, He-V927 #1 : Substrate

- CS-16-055, He-V927 #2 : Substrate

- CS-16-055, He-V927 #3 : N/A

- CS-16-055, He-V927 #4 : N/A
- CS-16-055, He-V927 #5 : N/A

- CS-16-055, He-V927 #6 : Substrate

- CS-16-055, He-V927 #7 : N/A

- CS-16-055, He-V767 #1 : Substrate
- CS-16-055, He-V767 #2 : Merged jets

- CS-16-055, He-V767 #3 : N/A

- CS-16-055, He-V767 #4 : Particle

- CS-16-055, He-V767 #5 : N/A (Horizontal Jet)
- CS-16-055, He-V767 #6: Substrate

- CS-16-055, He-V767 #7: Substrate

- CS-16-056, He-V927 #1: Substrate

- CS-16-056, He-V927 #2: Merged jets
• CS-16-056, He-V927 #3 : Merged jets

• CS-16-056, He-V927 #4 : Merged jets

• CS-16-056, He-V927 #5 : Substrate

• CS-16-056, He-V927 #6 : Merged jets
- CS-16-056, He-V927 #7 : N/A

- CS-16-056, He-V927 #8 : N/A

- CS-16-056, He-V927 #9 : Particle

- CS-16-056, He-V767 #1 : Merged jets
• CS-16-056, He-V767 #2 : Merged jets

• CS-16-056, He-V767 #3 : N/A

• CS-16-056, He-V767 #4 : Merged jets

• CS-16-056, He-V767 #5 : N/A
• CS-16-056, He-V767 #6 : N/A

• CS-16-056, He-V767 #7 : N/A

• CS-16-056, He-V767 #8 : Merged jets
Appendix 8: Surface Roughness

- Powder(0.3um x 0.3um)

- Powder(0.5um x 0.5um)

- Splat top(0.1um x 0.1um): CS-16-054A
• Splat top(30umx30um): CS-16-054A

![Image of Splat top(30umx30um): CS-16-054A]

• Mushroom(1.0umx1.0um): CS-16-055C

![Image of Mushroom(1.0umx1.0um): CS-16-055C]

• Mushroom(1.0umx0.25um): CS-16-055C

![Image of Mushroom(1.0umx0.25um): CS-16-055C]
- Mushroom(1.0umx1.0um): CS-16-055C

![3D image of Mushroom(1.0umx1.0um): CS-16-055C]

- Mushroom(1.0umx1.0um): CS-16-055C

![3D image of Mushroom(1.0umx1.0um): CS-16-055C]

- Mushroom(1.0umx1.0um): CS-16-056C

![3D image of Mushroom(1.0umx1.0um): CS-16-056C]
• Mushroom(2.0umx2.0um): CS-16-056C

• Mushroom(1.0umx1.0um): CS-16-056C

• Mushroom(2.0umx2.0um): CS-16-056C
• Mushroom (10.0um x 10.0um): CS-16-056C
## Appendix 9: Shear Test

<table>
<thead>
<tr>
<th>#</th>
<th>X-Vel.</th>
<th>Fz</th>
<th>Avg. Friction Force</th>
<th>Splat Dia.</th>
<th>Sheared Area</th>
<th>Shear Work</th>
<th>Shear Force</th>
<th>Shear Stress</th>
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<tr>
<td></td>
<td>um/s</td>
<td>N</td>
<td>N</td>
<td>um</td>
<td>um²</td>
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<td>-</td>
<td>3471.4</td>
<td>3.88E-05</td>
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Group, Sample, Splat No. : Before / After

- CS-16-055, He-V927, #1

- CS-16-055, He-V927, #30
- CS-16-055, He-V927, #32
  ![Image](image1.png)

- CS-16-055, He-V927, #19
  ![Image](image2.png)

- CS-16-056, He-V927, #2
  ![Image](image3.png)

- CS-16-056, He-V927, #3
  ![Image](image4.png)
• CS-16-056, He-V767, #1

• CS-16-056, He-V767, #2

• CS-16-056, He-V767, #3
## Appendix 10: Results of A-constant Study

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<thead>
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<th>A&lt;sub&gt;S&lt;/sub&gt;</th>
<th>Condition</th>
<th>Condition</th>
<th>Condition</th>
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<tbody>
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<td>270</td>
<td>Flat Top, Mixed Jets Bonding</td>
<td>Convex Top, Mixed Jets No Bonding</td>
<td>Convex Top, Substrate Jet Bonding</td>
</tr>
<tr>
<td>=A&lt;sub&gt;p&lt;/sub&gt;x1.00</td>
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<td></td>
<td></td>
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<td>337.5</td>
<td>Flat Top, Mixed Jets Bonding</td>
<td>Convex Top, Mixed Jets No Bonding</td>
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<td>405</td>
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Appendix 11-1: Simulation Results, Temperature 373K & 423K

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<td>100</td>
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<tr>
<td>150</td>
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<td><img src="image6.png" alt="Image" /></td>
</tr>
<tr>
<td>200</td>
<td><img src="image7.png" alt="Image" /></td>
<td><img src="image8.png" alt="Image" /></td>
</tr>
<tr>
<td>250</td>
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## Appendix 11-2: Simulation Results, Temperature 473K & 523K

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### Appendix 11-3: Simulation Results, Temperature 573K & 623K

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<td><img src="image9.png" alt="Image" /></td>
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Bibliography


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