

The Adhesion Strength of a Plasma Sprayed Silicon Bond Coating on a Silicon Carbide Ceramic Matrix Composite

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Abstract

Silicon-based ceramics and ceramic matrix composites (CMCs), such as silicon carbide (SiC) fiber reinforced SiC, are promising candidates for hot section components in next generation turbine engines. Environmental barrier coatings (EBCs) are essential for implementing these components as they insulate and protect the substrate from reaction with water vapor in the engine environment. EBCs are typically deposited via atmospheric plasma spraying (APS) and preparing the component surfaces through cleaning and roughening prior to coating is a vital step to ensure sufficient coating adhesion. The adhesion of a plasma sprayed coating to the underlying component is one of the most important properties as the component will not be protected if the coating is not well adhered. Surface roughening of metallic components via grit blasting is well documented and understood, but much less is known about preparing ceramic and ceramic composite surfaces for thermal spray coating. Silicon coatings are often used as a bond coating between SiC-based components and EBC top layers, but the adhesion strength of plasma sprayed Si on these substrates, Si splat formation and the factors that affect coating formation and adhesion have not been well studied.

The effects of automated grit blasting process parameters on surface roughness and material loss of a reaction bonded SiC (rb SiC) composite were evaluated. Surface roughness before and after grit blasting was evaluated with a confocal laser scanning microscope. The differences and advantages of automated grit blasting compared to manual grit blasting were observed. Most notably was the level of control at high nozzle traverse speeds resulting in reduction of material loss and consistency of roughening. At high nozzle traverse speeds, the amount of material loss decreased greatly with a small effect on induced surface roughness. The degree of grit blasting induced roughness and material loss was found to be largely dependent on the nature of the composite matrix and reinforcement, as well as blast nozzle traverse speed. A statistical model was developed to predict the substrate thickness loss and induced average roughness based on nozzle traverse speed and blast pressure for automated grit blasting.

Additionally, laser ablation was used to create controlled, regularly patterned surface texture on rb SiC substrates to further investigate the role of texture parameters in Si coating adhesion. Si was plasma sprayed onto rb SiC substrates to deposit both thick coatings to evaluate adhesion strength and single splats to study splat formation. Surface roughness/texture, substrate preheat temperature and mean Si particle size were varied in plasma spray coating experiments to observe their role in coating adhesion strength. Si adhesion strength was found to be related to all three factors and a statistical model was developed to predict adhesion strength based on them. Substrate preheat temperature had a significant effect on both Si adhesion strength and Si splat formation on rb SiC.

Single splat formation during plasma spraying of Si on SiC was simulated with software called SimDrop. Simulations of Si droplet impact, spreading and solidification during plasma spraying on smooth and textured SiC surfaces were used to investigate the effects of relevant process parameters on splat formation. Experimentally observed Si splats on smooth substrates at different temperatures during deposition were matched with simulated splats with the same spraying parameters. A change in thermal contact resistance with changing substrate preheat temperature was confirmed by the simulation results. The role of surface texture parameters for a regularly patterned surface texture in splat formation was demonstrated through simulation.

This dissertation investigates methods of roughening and preparing a SiC composite substrate for plasma spray coating, as well as factors which affect the adhesion strength and splat formation of plasma sprayed Si through experiments and simulation. The observations made provide valuable insight for understanding and optimizing the manufacturing processes utilized to deposit strongly adhered coatings onto SiC-based composites. In addition, areas of interest in this field for future study and further investigation are introduced and suggested.

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1 Introduction

As improved performance and efficiency is constantly desired in any engineering field, new technologies arise, and different materials and processes are utilized to solve problems. Specifically, in the aerospace industry, the demand for higher engine operating temperatures, engine weight reduction and longer component life span pushes the development of new material systems, coatings and coating methods. The use of ceramic materials, particularly SiC/SiC ceramic matrix composites (CMCs), for components in high temperature and high-pressure sections of aerospace propulsion engines has recently been of strong interest for engine manufacturers. The low density, high temperature capability and resistance to oxidation and corrosion makes these materials ideal for hot section components. Utilizing these materials would greatly reduce engine weight and the need for cooling during operation. However, there are still challenges to be faced with implementing CMCs because they are a relatively new material system, and the vast amount of knowledge associated with metal materials cannot necessarily be applied to these composites. One such challenge is optimizing the coatings which must be applied to CMCs for use in jet engines, and this includes optimizing surface preparation prior to coating.

Coatings have been used in a variety of industries for ages to improve several properties of the underlying material. In more recent years, thermal spray coatings are commonly used on components implemented in harsh environments to improve wear resistance, corrosion resistance and maintain overall surface and mechanical properties. Coatings for aerospace applications are typically deposited via atmospheric plasma spraying (APS). A primary challenge in this area currently is adhesion of the plasma sprayed coating. Adequate adhesion of the coating to substrate/component is required for proper coating performance. Substrate surface preparation prior to coating is critical to coating buildup during spraying and coating adhesion [1-3]. It is well known that a clean, rough surface is vital for coating adhesion. A roughened surface has a surface profile which facilitates a mechanical interlocking of the coating with the substrate. Prior to coating, surfaces are typically cleaned and then roughened with grit blasting, although there are a variety of surface preparation methods used for the purpose of surface roughening, including high-pressure water jet, dry ice blasting, laser surface texturing and others [1].

While it is fully accepted that surface cleanliness and roughness are important for coating adhesion strength, there is no strong agreement about the nature of roughness or topography which optimizes adhesion strength. R_a is often reported to be related to the important property, coating adhesion [2, 4, 5], but some studies in the literature also report that R_a is not necessarily indicative of adhesion strength [3, 6, 7]. The nature of coating buildup during spraying ultimately determines coating adhesion/cohesion, but the initial coating droplet impacts, or splats, on the substrate surface is especially important for generating a strong bond between coating and part. Many factors can affect the behavior of coating droplets during plasma spraying, including substrate surface properties, sprayed particle properties and parameters of the actual plasma spray process. Understanding the role of surface texture and state in the initial droplet impact during coating is one large piece of the puzzle of optimizing coating adhesion and performance.

This research looks to explore methods of preparing SiC surfaces for plasma spray coating, as well as to better understand and optimize the adhesion strength of a plasma sprayed Si coating. Of particular interest is the role of various surface roughness parameters in coating behavior and adhesion strength. As grit blasting is the primary technique used for preparing parts before coating, the ability to control the resulting surface through grit blasting is greatly desired. However, almost no work has been reported on the effects of grit blasting of SiC, neither the material/thickness loss due to grit blasting nor the adhesion strength of plasma sprayed Si coatings. This work shows how different grit blasting process parameters can be adjusted to alter surface roughness and/or thickness loss induced on reaction bonded silicon carbide (rb SiC). Laser ablation was also used to generate surface texture patterns on rb SiC and to study the role of surface texture parameters in plasma sprayed Si coating adhesion strength. The effect of Si mean particle size and rb SiC substrate preheating on coating adhesion strength was also investigated, along with the effect of substrate temperature on plasma sprayed Si single splat formation. Finally, the impact, spreading and solidification of a plasma sprayed Si droplet on a SiC surface was studied via simulation by varying impact parameters based on experimental measurements of plasma spraying process parameters, and experimental observations were compared with simulation results. The effect of patterned surface texture on Si splat formation was also modelled by utilizing simulated surface texture similar to that created on substrates by laser ablation.

2 Theoretical Background

2.1 Ceramic Matrix Composites (CMCs) and Environmental Barrier Coatings (EBCs)

The use of nickel-based superalloys for some engine components has begun to be a limiting factor in improving efficiency as operating temperatures have reached their maximum for this material. CMCs, which consist of a ceramic matrix and a reinforcement of some kind, such as SiC fibers in a SiC matrix, are of interest for implementation in aircraft and are planned for use in the next generation engines. SiC/SiC CMCs provide the benefit of low density, higher temperature capability and oxidation resistance, and are of strongest interest to engine manufacturers because of these properties which allow for higher engine operating temperatures and a significant reduction in weight. However, these materials are still vulnerable to reaction with water vapor in the engine environment [8,9]. Environmental barrier coatings (EBCs) are required for implementation of CMCs as hot-section components in aircraft propulsion engines, and these coatings are typically deposited via atmospheric plasma spraying (APS). The substantial advantage in terms of temperature capability which both the use of SiC/SiC CMCs and EBCs provide is clearly illustrated in Figure 1. The novelty of CMCs for these applications in aerospace means that not much is known about how to optimize the coating application and substrate surface preparation processes for the specific materials involved. The adhesion strength of these EBCs and any associated bond coating is of primary concern and there is much room for improving overall understanding in this area.

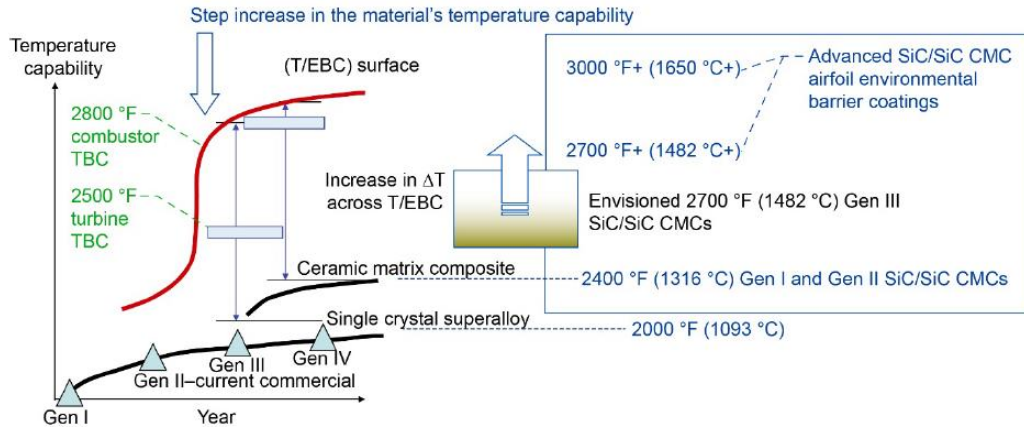


Figure 1. The impact of the implementation of SiC/SiC CMCs and plasma sprayed environmental barrier coatings (EBCs) on engine temperature capability [8]

2.2 Atmospheric Plasma Spraying

Thermal spray coatings are commonly used on components implemented in harsh environments to improve wear resistance, corrosion resistance and maintain overall surface and mechanical properties. APS is one type of thermal spraying in which the desired coating material, typically in powder form, is melted and accelerated towards the substrate via a plasma torch in open air. A high intensity, direct current arc between an anode and cathode nozzle is generated as a mixture of gases, usually hydrogen and argon, flows through to create the high energy plasma. The plasma jet can reach temperatures well above 7000°C, which allows for melting and coating generation with almost any material [1]. Individual particles of the desired coating material travel towards the substrate at high velocity after being melted, impact a usually roughened substrate surface, spread on the surface and solidify to create a splat. The process of plasma spraying and a few of the many variables involved are illustrated in Figure 2. The buildup and layering of splats during this process results in a full coating. As these splats are the building blocks of the whole coating, understanding the behavior of individual splats during spraying and the role of process parameters which may affect this behavior is of great importance. The adhesion of the coating to the underlying material is caused by mechanical

interlocking with the substrate surface features after solidification; and the first splats to impact during coating are likely to determine the adhesion strength at this interface [1].

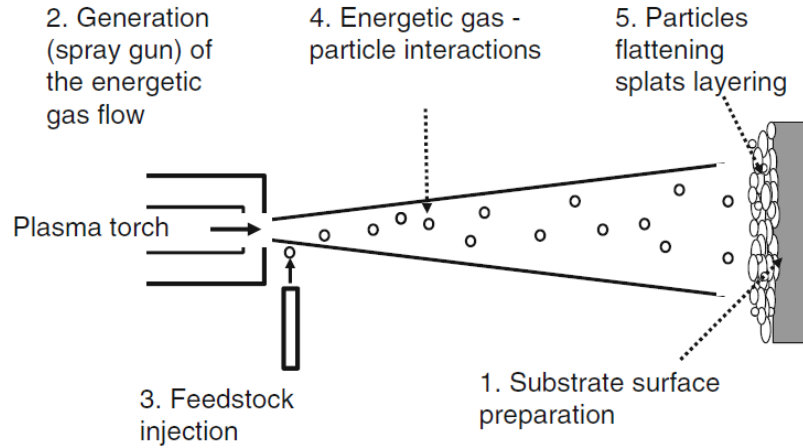


Figure 2. Schematic of the major components of the plasma spray process [1]

2.2.1 Thermal Spraying of Silicon

Silicon coatings are often used as a bond coating between SiC-based components and EBC top layers because it can serve as a protective layer as it readily forms an oxide layer which helps prevent oxidation of the underlying substrate [10-13]. The Si bond coat is also used to improve the adhesion between the substrate and upper coating layers by acting as an intermediate in terms of thermal expansion coefficient [14,15]. Depositing Si coatings via APS is more cost effective than techniques like physical or chemical vapor deposition (PVD or CVD), and APS is also used for depositing EBCs. Thermal spraying of Si coatings has been fairly well studied [10, 16-19], especially for electronic applications [20, 22-30]. However, only a few studies have focused on thermal spraying of Si onto SiC-based substrates with the use as a bond coating in mind [10,19]. For electrical applications, Kharas et al. [20] used a non-commercial plasma torch to deposit Si with a mean particle size of 13.8 μm on thermally oxidized Si wafer substrates to characterize the morphology and microstructure of splats and deposited films. Niu et al. [18] deposited Si on titanium substrates via APS and characterized the composition and microstructure of the sprayed coating. APS was used to coat a titanium alloy with Si and the Si powder size was varied to study the effect on in-flight particle properties, splat morphology and

coating microstructure and properties [17]. It was found that in-flight particle velocity, temperature and oxidation increased with decreasing particle size. Nistal et al. [10] produced dense coatings of Si on SiC substrates via flame spraying (FS), which reaches a much lower flame temperature and particle velocity compared to APS, and characterized the deposited coating material and thermal properties.

One of the most important properties of a coating, especially in extreme and harsh environments, is how well it stays attached to the underlying substrate, which is often characterized by the bonding or adhesion strength, because it must be well adhered to the substrate to protect it. Only one of the studies [16] on thermal spraying of Si reported the resulting coating bonding strength on Ti alloy substrates, however effects of preparation or spray parameters on bond strength were not reported. The measured bonding strength was reported to be 20.6 ± 1.9 MPa, or 2988 ± 275 psi. As Si is heavily considered as a bond coating for EBCs, evaluating its bonding strength on relevant substrates and the factors that affect it is vital for optimizing these bond coatings and EBC systems.

2.3 Substrate Surface Preparation

The cleanliness, roughness and overall state of the substrate surface to be coated is extremely important as these factors will ultimately affect the adhesion of any plasma sprayed coating. This is where substrate surface preparation prior to coating plays a key role. Many different methods are used to clean and/or roughen a surface, such as substrate preheating, ultrasonic cleaning, dry ice blasting, grit blasting and laser treatment. The appropriate cleaning and roughening method to be used will depend on the coating and substrate materials, but a clean, rough surface is ideal for ensuring a strong bond between coating and substrate.

2.3.1 Substrate Preheating

The role of substrate preheating and temperature in plasma sprayed coating formation and adhesion strength has been well studied for a range of coating and substrate materials, primarily metal substrates [31-44]. As most surfaces exposed to atmosphere will have an adsorbate or condensate presence, particularly when an oxide layer exists on the surface [1], and heating the

substrate can act to clean the surface by evaporating and desorbing the adsorbed species. Additionally, preheating substrates will affect the formation of an oxide layer on surfaces which an oxide forms like metals and SiC. The method used to preheat substrates, whether the atmosphere is controlled, the rate and evenness of heating and exposure to the flame/plasma jet, will also play a role in oxide layer formation and surface effects [1]. While oxide growth and surface effects of heating of many materials via conventional methods has been well studied, only a few studies on metal substrates have been conducted on the effects of plasma jet preheating on oxidation and surface composition [1, 45-48].

Bianchi et al. [31] reported that the adhesion-cohesion values of plasma sprayed yttria-stabilized zirconia (YSZ) coatings were three times higher on stainless steel substrates preheated to 300 °C by a radiative heating system compared to non-preheated substrates regardless of in-flight particle parameters. It was also found that coating adhesion-cohesion initially increased with preheating time before reaching a maximum and then decreasing for longer preheat times when the oxide layer was likely too thick. Another author [44] reported on plasma sprayed nickel coatings on stainless steel substrates preheated to various temperatures via a block heater and found that the coating adhesion strength increased significantly with substrate temperature, going from 10 MPa at 25 °C to 74 MPa at 650 °C. Coating porosity was also seen to decrease with increasing substrate temperature. Substrate oxide layer thicknesses increased with increasing preheat temperature and corresponded to greater adhesion strength; however when stainless steel substrates were heated to 650 °C, held for 2 min to form a thick oxide and then allowed to cool before being coated, the average adhesion strength was only 14 MPa. In addition, cobalt alloy substrates much less susceptible to oxidation were coated with varied preheat temperature and coating adhesion strength increased from 16 MPa at room temperature to 55 MPa at 500 °C. This likely indicates that the substrate being held at elevated temperature during coating deposition is important for improving adhesion and cannot be attributed to the formed oxide layer.

Fukumoto et al. [36] also reported that the adhesion strength of nickel coatings plasma sprayed on AISI304 stainless steel substrates increased with substrate preheating. It was observed in another study [32] that increasing substrate temperature during plasma spraying of molybdenum on steel, molybdenum and glass substrates resulted in improved coating mechanical properties, porosity and thermal conductivity. Yang et al. [38] found that the

adhesion strength and hardness of plasma sprayed copper coatings on stainless steel substrates increased when the substrate temperature was increased via longer preheating times with the plasma torch prior to coating.

2.3.2 Surface Roughness and Coating Adhesion

The topic of the relationship between surface roughness and plasma sprayed coating adhesion is a complex one. It is generally accepted that a roughened substrate shows improved coating adhesion as compared to a smooth substrate, although, as mentioned previously, there have been mixed reports of the effect of average surface roughness on plasma sprayed coating adhesion strength. Due to the complexity of both the topography of a surface and the plasma spray coating process, it is challenging to isolate and study the variables which play a role in coating adhesion. Only a few authors have been able to find an indication of a relationship between roughness parameters of grit blasted substrates beyond R_a and plasma sprayed coating adhesion strength [6, 49-50]. Bahbou et al. [49] and Bobzin et al. [6] reported that the parameter $R_{\Delta q}$, which describes both amplitude and spacing properties of a surface profile with the slope of roughness, had a stronger relationship with coating adhesion strength than R_a . In a different study, Bahbou et al. [51] investigated the relationship between fractal dimension of grit blasted substrates and plasma-sprayed coating adhesion, along with R_a , and found a poor correlation between fractal dimension and adhesion strength. A much stronger correlation was found between R_a and adhesion strength in this study. Griffiths et al. [50] also showed results indicating there is a relationship between the peak slope and coating adhesion strength. There was also seen to be a relation between peak spacing and adhesion in which the adhesion strength increased with peak spacing up to an optimum around 200 μm and decreased thereafter. Intuitively, this makes sense as it would be expected that surface asperities must be packed close enough to allow a strong interlocking, but also must not be spaced so closely that the coating can't penetrate the rough surface. However, all of these studies were performed on metal substrates and no known work has been done to repeat or confirm the findings.

A few authors have studied the effects of surface texture generated via laser surface texturing technique on coating adhesion and behavior [52-55]. The results of these studies indicate that the size of surface texture features play a large role in coating adhesion and the substrate/coating interface, especially in relation to the size of the impacting droplets during coating. Three different texture patterns, a groove, square and hexagonal pattern, were generated

via laser texturing and it was found that the plain or untreated area in between laser generated surface texture was detrimental to the adhesion strength of a plasma sprayed coating [54]. Tan et al. [53] generated dimple patterns with varying dimple diameters and distances on steel substrates and subsequently coated them with a nickel coating via APS. The dimple distance was seen to have a more significant effect on coating adhesion strength than dimple diameter, and adhesion strength tended to increase with increasing diameter-to-distance ratio. The sample texture with the highest diameter-to-distance ratio and coating adhesion strength consisted of simply peaks/posts on the surface due to the high dimple distance, which likely corresponds to reduced plain area on the surface compared to other textures. Kromer et al. [56] found that Ni-Al coatings plasma sprayed onto laser textured aluminum alloy substrates exhibited stronger adhesion than coatings on substrates which were grit blasted via conventional methods. Optimizing the laser-generated hole volume for the size of sprayed particles allowed for the elevation of the coating/substrate adherence. A flame sprayed alumina coating was deposited on a SiC/SiC CMC previously laser treated to generate peaks on the surface [57]. The laser treatment modified the surface texture as well as the surface chemistry enhancing the adherence of the alumina coating.

2.3.3 Grit Blasting

Substrate surface preparation prior to coating is critical to coating adhesion strength, and adequate adhesion is required for proper coating performance [1]. Grit blasting is the most commonly used method for roughening a surface before coating, and it produces a complex surface topography which can be difficult to fully describe. During this process, abrasive particles are accelerated towards the substrate via either a suction or pressure type blast machine. The abrasive grit media used is typically a hard ceramic like alumina, silica or silicon carbide, and upon high speed impact of the particles with substrate, roughness is generated due to material removal via cutting, brittle fracture and plastic deformation. The grit to be used will be determined by the properties of the material being grit blasted, as this will affect material removal, along with desired roughness. This surface roughening increases overall surface contact area and creates prominent asperities on the surface that allow the coating to penetrate and lock into, improving adhesion strength. The type and degree of roughness for promoting adhesion of a

plasma spray coating is still debated, and there is still substantial room for improved understanding of this topic.

Grit blasting is primarily done manually, without strict control over blast parameters. These blast parameters, such as pressure, angle, grit mass flow rate, standoff distance, nozzle traverse speed, grit material and grit size are known to affect the resulting roughness of the blasted surface [1-3, 6, 51, 58]. Figure 3 shows a diagram of a standard grit blasting setup, whether automated or manual, with the blast angle relative to the substrate surface and standoff distance measured from nozzle exit to substrate. The capabilities and differences associated with the use of an automated robot during blasting are much less documented, but of strong interest

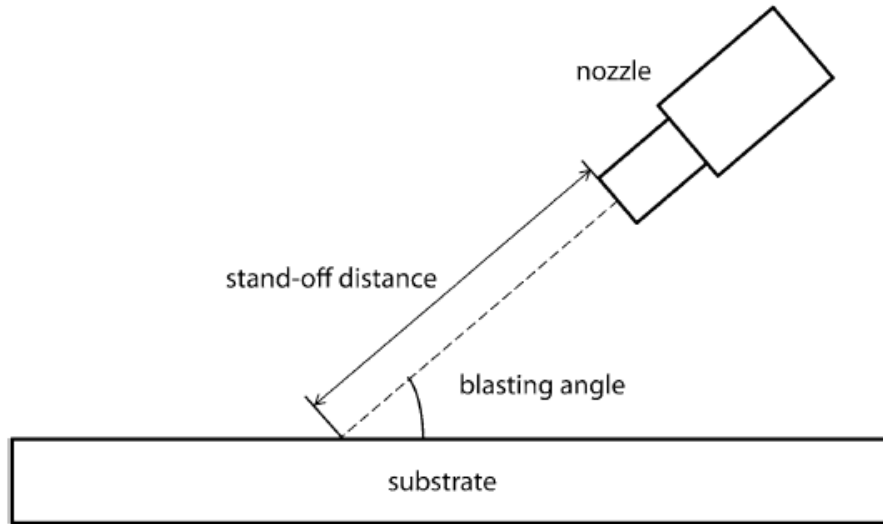


Figure 3. Typical experimental setup for grit blasting [6]

for improving the consistency of the process.

Much is known about how the grit blasting process parameters affect metallic substrates in terms of roughness [1-6, 49-51, 58-61], but there is still a lack of knowledge related to the effects of grit blasting on ceramic and ceramic matrix composite materials (CMCs) due to the recent rise in interest in coating and implementing these materials. As there is a strong interest in utilizing ceramic-based composites in increasingly high temperature and corrosive environments, it is vital to understand every step of the process of preparing and coating these materials. It is possible that the effects of grit blasting parameters on the induced roughness of a SiC surface will be similar to that of metallic substrates, but it is important to perform experiments to obtain a complete and confident understanding. The grit blasting process has many variable parameters

which all affect the final surface, and the results will likely always depend on the materials being used. Thus, there can always be improvements made to the process in general, and for a specific material and desired surface topography. Few authors have reported methods of controlling roughness parameters beyond the average height roughness R_a or independent of R_a . The ability to manipulate the size of surface asperities based on the size of the impacting coating particles would be of tremendous benefit in order to facilitate the penetration of the coating into the surface. This is an area which requires further study in order to realize the capabilities grit blasting may provide for optimizing coating adhesion strength.

A concern associated with roughening a surface via grit blasting is the effect it may have on the surface and mechanical properties of the substrate. Grit blasting can remove a relatively large amount of material, and this must be planned for when preparing a surface for coating. Reducing material removal and the effects on surface/mechanical properties while creating a surface which promotes mechanical interlocking during thermal spray coating is ideal. The effect of grit blasting and specific grit blasting parameters on degree of material removal is rarely ever reported in literature, but this is important to consider, especially for parts that have tight dimensional tolerances, have a protective top surface layer or are more sensitive to grit blasting.

2.3.4 Laser Ablation

Another surface preparation method which is of strong interest in the world of thermal spray coating is laser ablation. In this process, the substrate material is irradiated with a high-power laser for a short duration, resulting in vaporization, melting and/or ejection of material near the surface [62]. This technique is thus very useful for surface preparation that could include cleaning, heating or generation of surface texture before coating. Depending on the substrate material, the laser system used and the desired result from laser ablation, process parameters such as laser power, pulse duration, wavelength, spot size and number of pulses can be varied to affect the surface in different ways [56]. Cleaning or heating the surface for example will likely require different laser treatment parameters than removing material to create a desired surface topography or texture. An advantage of laser ablation over a method like grit blasting is greater control over the final surface. A small spot size allows for very small areas of a surface to be treated, and laser systems can be programmed to generate specific patterns on the surface and create much more controlled roughness. This makes studying the effects of surface roughness

and specific topographical features much easier compared to the chaotic and random roughness generated with grit blasting. Figure 4 illustrates the capability laser ablation provides for obtaining texture patterns on to-be-coated surfaces. Beyond simple hole patterns like that of Figure 4 and the ability to strictly control hole dimensions like diameter, depth and spacing, laser ablation can be used to create angled texture, groove patterns and any variety of surface features which may be used to alter the degree of mechanical interlocking obtained between the coating and the substrate.

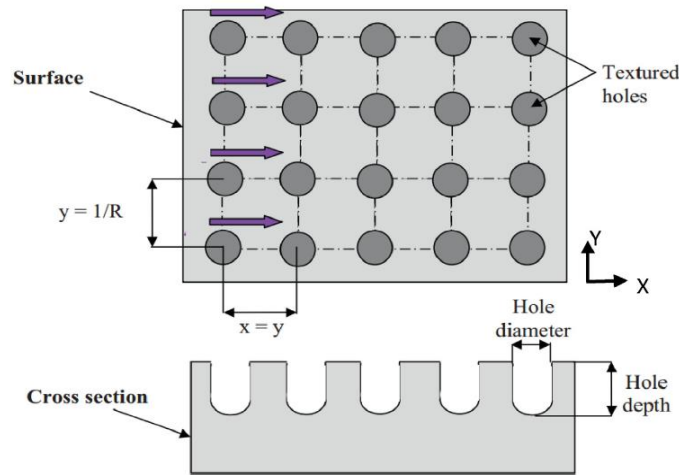


Figure 4. A schematic of one type of surface texture pattern which can be obtained via laser ablation of a surface [56]

2.3.5 Roughness Parameters

Characterization of the surface state and topography after grit blasting is important for understanding the nature of coating adhesion and the coating/substrate interface. Gademawla et al. [63] presents in detail the measurement and definition of 2D roughness parameters. Typically, only a few roughness parameters, or simply the average roughness R_a , are used to describe a grit blasted surface. R_a isn't a sufficient description of surface topography, especially when it comes to surface-coating interaction [49]. R_a provides a generic description of the average roughness of a surface and is therefore most commonly used. However, it is not able to capture the intricate details of the surface, such as if there are deep valleys, sharp peaks or large spaces between peaks, which could all affect coating behavior and adhesion. As R_a is just an average, two

surface profiles could have the same value of average roughness but be extremely different in a vast number of ways, as seen in Figure 5. Using only R_a to evaluate roughness will not capture these differences that can exist in surface profiles, especially after grit blasting. R_a is often reported to be related to the important property, coating adhesion [2, 4-5], but some studies in the literature also report that R_a is not necessarily indicative of adhesion strength [3, 6-7]. Roughness parameters beyond R_a give a more complete description of surface topography but have not been studied in depth in relation to coating adhesion. The height of peaks (R_p) and depth of valleys (R_v) likely play a significant role in how deep a coating penetrates into the surface and affect the quality of mechanical anchoring [1]. Other parameters, such as peak spacing (R_{sm}), peak slope ($R_{\Delta q}$) and reduced valley depth (R_{vk}), have been studied by other researchers and found to affect coating adherence on metal substrates [6, 49-50]. The shape and size of surface asperities, peaks and valleys described by these non- R_a parameters must also be considered in relation to the size of particles of coating material. Griffiths et al. [50] found that the optimum peak spacing for plasma sprayed alumina coating adhesion strength depended on sprayed particle size, and Kromer et al. [52] reported similar findings.

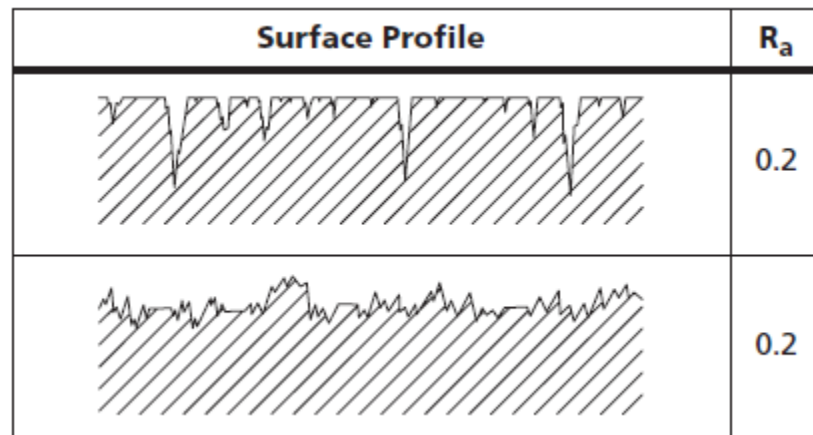


Figure 5. Two very different surface profiles with the same average roughness R_a [64]

R_a (arithmetic mean height deviation) describes the average height roughness of the profile and is the most commonly reported parameter when describing the roughness of a surface. R_z (average peak to valley distance) is a measure of the distance between the highest peak and lowest valley within each sampling length and is averaged from all sampling lengths.

R_v (maximum valley depth) describes the average depth of the lowest valley from all sampling lengths and R_p (maximum peak height) gives the average of the highest peaks from each sampling length. R_{sm} (mean width of profile elements – peak spacing) describes the spacing between profile elements or peak spacing and likely plays a large role in the ability for coating material to penetrate into the valleys and between peaks of the surface. $R_{\Delta q}$ (root mean square slope – surface slope) describes the average slope of the profile, and therefore profiles both amplitude and spacing information. These 2D roughness parameters are reported and defined according to EN ISO 4287 [65] as:

$$Ra = \frac{1}{l} \int_0^l z(x) dx$$

where l is the measurement length and z is the surface height.

$$Rv = \min (z(x))$$

$$Rp = \max (z(x))$$

$$Rz = \frac{1}{n} ((Rp1 - Rv1) \dots + (Rpn - Rvn))$$

Where n is the number of sampling lengths.

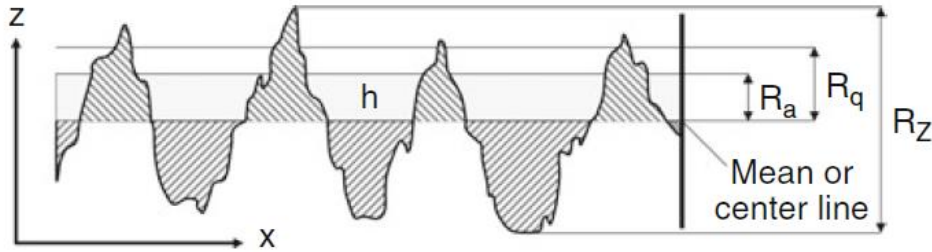


Figure 6. Diagram of common parameters used to describe the roughness of a surface profile [66]

$$Rsm = \frac{1}{n} (Xs1 \dots + Xsn)$$

Where X_s is the width of profile elements or space between peaks.

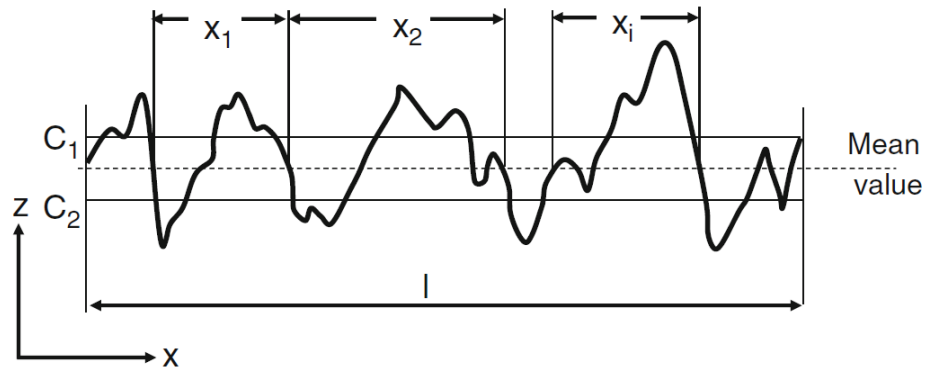


Figure 7. Definition of the mean width of profile elements or peak spacing R_{sm} (99)

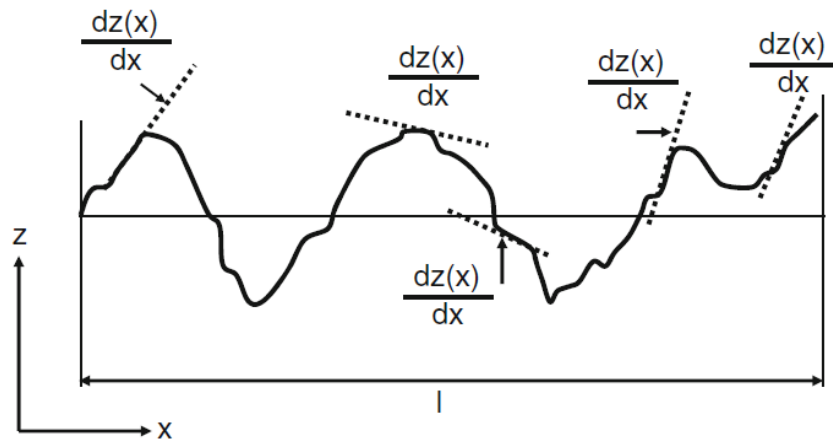


Figure 8. The slopes of a profile used to calculate Rdq [49]

$$R\Delta q \text{ (or } Rdq) = \sqrt{\frac{1}{l} \int_0^l \left(\frac{dz}{dx} \right)^2 dx}$$

2.4 Splat Formation in Plasma Spraying

2.4.1 Effect of Process Variables

Any thermal spray coating process consists of the acceleration of molten or semi-molten particles towards a substrate and the subsequent impact, spreading and solidification of those individual droplets on the surface to form splats. The final coating is made up of many of these solidified splats each having their own impact and spreading events that determine the resulting coating properties. There are a large number of variables associated with any thermal spraying process that play a role in splat formation. The substrate and coating material properties, substrate and particle temperature, particle velocity, impact angle, substrate and particle oxidation, the presence of adsorbates or condensates on the substrate surface and the thermal contact resistance between impacting particle and substrate will all affect the splat formation.

Bianchi et al. [31] reported that plasma sprayed YSZ splats on stainless steel were more lenticular at lower impact velocities, however splat shape also depended on substrate temperature. It was also found that splat thickness increased with decreasing impact velocity and increasing particle diameter. The YSZ splat cooling rate increased with increasing particle impact velocity and temperature, however a lower temperature or oxidized substrate greatly reduced the cooling rate in all cases. Leger et al. [67] observed similar behavior as the cooling rates of zirconia splats increased with particle temperature and velocity. Observations by Li et al. [68-69] showed that splat splashing increased with particle kinetic energy, represented by the Reynolds number, even when the substrate was sufficiently preheated. Researchers [70-71] showed that the substrate transition temperature required to form disc splats was dependent on substrate and spray material, as well as substrate thermal conductivity. The transition temperature increased with increasing substrate thermal conductivity. Nistal et al. [10] reported that the lower adhesion strength of flame sprayed Si on SiC was related to reduced particle temperature and velocity at a greater standoff distance. The oxidation of thermally sprayed particles, especially with the high temperatures associated with plasma spraying, is important to consider as it relates to splat formation and coating properties [1]. Studies [10, 17, 20] pointed out that oxidation of Si particles during thermal spraying likely affect splat to substrate and inter-splat bonding.

2.4.2 Effect of Roughness

The buildup of a coating during the plasma spray process, coating properties and ultimately coating adhesion strength greatly depends on the initial interaction between the particles being sprayed and the substrate, which is affected by the surface topography and morphology [50, 52]. The spreading of a molten particle, the filling of surface voids and interfacial contact area upon impact will be partially controlled by the frictional forces on the surface created by the roughness, and more specifically by the amplitude and spacing of peaks [72], as illustrated in Figure 9. Two primary types of coating splat morphologies have been found to be associated with both coating adhesion and substrate surface topography. Disc splats are favorable and correspond to higher adhesion strength, while splash splats are not favorable and correspond to more voids at the interface between coating and substrate, and thus lower adhesion strength values [50]. The topography and morphology of the surface where the splat forms are very important in determining which type of splat will form. When peak/valley spacing is not optimized for particle size, a higher number of splash splats will likely be observed, resulting in less contact area at the interface between substrate and coating, and decreasing adhesion strength. Roughness is known to result in reduced spreading of splats compared to a smooth surface, but the question of the optimum nature of roughness for improved contact area and adhesion strength is still to be answered.

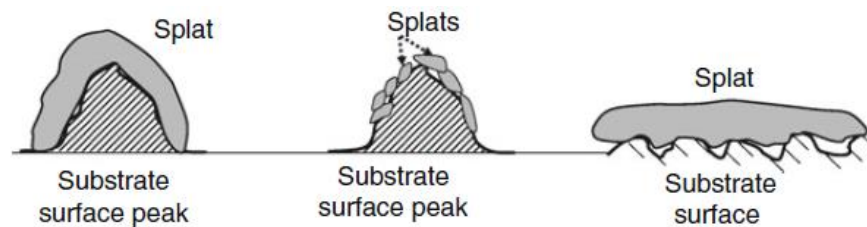


Figure 9. Diagram showing the surface filling and interfacial contact area of splats on surfaces with varied peak sizes [1]

Bianchi et al. [31] observed a reduction in YSZ splat flattening degree and increase in splat thickness with increased substrate surface roughness. The splat cooling rate also decreased with increased roughness due to the increased splat thickness and reduced splat/substrate contact. Similar results were found for molybdenum splats deposited on glass and molybdenum substrates [73] as increased surface roughness reduced splat flattening and flattening time. Shinoda et al. [74] plasma sprayed alumina onto micro-patterned quartz substrates and observed

the effect of surface texture on splat spreading and morphology. Concave and convex surface patterns resulted in splat fingering and air entrapment under deposited splats was seen for all texture patterns.

In addition to surface topography, many other factors affect the behavior of splats upon impact which must be considered when studying these phenomena. In particular, adsorbates and condensates on the surface, as well as the thermal contact resistance have been seen to play a significant role in the type of splats which form. Preheating metal substrates above a critical temperature prior to coating is considered to result in the removal of adsorbates and condensates on the substrate surface, ultimately increasing the generation of disc splats as opposed to splash splats during spraying.

2.4.3 Effect of Substrate Temperature, Adsorbates and Condensates

Many studies have looked into and observed the significant effect of substrate temperature on plasma sprayed splat formation [31-44, 70, 75]. A few explanations have been given for the splat transition behavior from splashed and distorted splats to coherent, disc splats that also corresponds to increased adhesion strength when substrate temperature is elevated during plasma spraying. Slowing of splat cooling due to higher substrate temperature and/or a thermally grown oxide layer which increases thermal contact resistance, increased wettability and nanoscale roughness due to oxide formation and the removal of adsorbates and condensates from the surface have all been proposed as the reasoning for the effect of substrate temperature on splat behavior. It is very possible that a combination of these factors contributes to the observed behavior and that variations in the substrate and coating materials, as well as in the spray process parameters, play a role.

Tran et al. [43] observed that the presence of hydroxide on the surface of an aluminum substrate was related to plasma sprayed NiCr splat breakup. When the role of preheating and surface treatment in surface roughness variation and splat behavior was studied [42], no significant difference in roughness was observed for preheated and non-preheated aluminum substrates on which NiCr was plasma sprayed, though splat morphology improved with substrate preheating. This suggests that splat formation transition is not caused by a change in surface roughness with preheating. When substrates were preheated, the formation of disc splats was

enhanced, and this corresponded to the conversion of hydroxide to oxide and removal of water vapor from the surface prior to spraying. Fukumoto et al. [36] varied stainless steel substrate temperature and ambient pressure independently during plasma spraying of Ni and found that splat transition behavior was similar for increasing substrate temperature and decreasing ambient pressure. Substrates were kept at room temperature while ambient pressure was varied and the sprayed splats still transitioned from splash to disc-shaped when pressure was reduced, indicating that a change in the surface state caused by reducing ambient pressure or increasing substrate temperature was responsible. In addition, the splat cooling rate was reduced with both increasing substrate temperature and decreasing ambient pressure. This strongly points to the removal of adsorbed gases, water or other contaminants on the surface causing the change in splat behavior. The results shared by Pershin et al. [44] in which plasma sprayed coating adhesion strength was significantly increased when substrate temperature was elevated during spraying but not when the substrate was heated to high temperature to form a surface oxide and then cooled before coating deposition also seems to indicate that the improved deposition behavior is more linked to removal of adsorbed species on the surface than to oxide layer presence. The improvement in splat morphology was observed when the coating method was changed from APS to vacuum plasma spraying by Sampath et al. [33,76], which also suggests an effect of surface adsorbates/condensates.

2.4.4 Simulation of Splat Formation

Many authors [77-84] have utilized models to study droplet impact and splat formation during thermal spraying, but few have looked at a rough surface. Parizi et al [85](27) used SimDrop software, which is based on the models developed by Bussman et al. [79] and Pasandideh-Fard et al. [82] to simulate the impact and solidification of liquid nickel and zirconia on textured silicon substrates. Solving the full Navier-Stokes equation, including heat transfer and phase change, the computational fluid dynamics software also utilizes a volume of fluid (VOF) algorithm to track the droplet-free surface. The simulations showed the effect of surface texture pattern parameters as well as thermal contact resistance and solidification on splat behavior upon impact. Surface texture was seen to affect the coating/substrate contact area, porosity and the nature of spreading of the splat. Experiments showed good agreement with the

simulation results in terms of splat behavior, splat size and presence of porosity. The spreading of plasma sprayed YSZ particles on concave, dimple-patterned substrates was studied numerically and experimentally [86]. The concave texture patterns on quartz substrates caused fingering and splashing of the impacting particles and the numerical simulation revealed that fingering and splashing occurred when the droplet viscosity was reduced. It was also observed that air between the dimples and the impacting droplet affected the spreading. Raessi et al. [87] simulated plasma spraying of alumina on an alumina substrate with a smooth surface and regularly spaced, cube-shaped surface peaks of varying size. They reported that solidification played an important role in splat shape and changes in the pattern peak size affected droplet spreading.

2.5 Simulent Software

A software product made by Simulent, Inc. was identified as a modeling software of interest for simulating droplet impact, spreading and solidification during the thermal spray process, along with coating build up and properties. SimDrop is capable of modelling textured surfaces, multiple droplet impact and manually setting material properties [88].

The 3D free surface code of SimDrop can determine the fate of single/multiple particles on the substrate during plasma spraying, find out the shape of the splat, solidification, and whether there is any splashing or not. The main inputs to this code are initial size, temperature and velocity of the molten droplets. This information can be obtained from experiments or desired values can be used. The user can also define the material properties of the substrate and the droplets to match the specific material system of interest, but droplet temperature and state, as in solid or liquid, are assumed to be uniform just before impact with the substrate. Droplet velocities up to 400 m/s can be modeled with the software, which is well above the velocities used in thermal spraying of current coatings of interest. SimDrop can model the impact of multiple droplets which are either randomly generated by the software or manually set by the user. This is of interest to gain an understanding of the droplet behavior when impacting a previously formed splat and how the coating is built up via individual splats, in addition to simply the surface of the substrate. The software allows for simulation of a textured surface in

which peak-to-valley height, peak-to-peak distance and peak width can be set to observe the effect of this texture on droplet impact and solidification. While the simulated surface may not have random surface texture like that of a grit blasted sample, very similar parameters can be implemented and studied with the software. In addition, laser ablation will allow for generating comparable texture on the surface of specimens, which can be coated and compared to both the model and grit blasted surfaces.

The screenshot shows the SimDrop software interface with the following sections:

- 1. Please specify the geometry of the surface :**
 - ☒ Flat ☐ Inclined
- 2. If the surface is inclined, please give the angle of inclination, Phi. To determine the value of Phi, please click on Help.**
 - Phi [degree]: 0.0
- 3. Is the substrate smooth or rough?**
 - ☒ Smooth ☐ Rough
- 4. Please give the roughness parameters (click on Help for more information):**
 - 4.1. Peak to valley, microns: 0.0
 - 4.2. Peak to peak, microns: 0.0
 - 4.3. Width, microns: 0.0
- 5. Is the substrate rotating?**
 - ☐ Yes ☒ No
- 6. Direction of rotation when looking from the top of the substrate:**
 - ☒ Clockwise ☐ Counter Clockwise
- 6.1. Rotational speed, rpm:** 0.0
- 6.2. X: position of center of rotation, m:** 0.0
- 6.3. Y: position of center of rotation, m:** 0.0

Figure 10. The SimDrop interface in which surface texture parameters peak to valley distance, peak to peak distance and peak width can be set for droplet impact simulation [88]

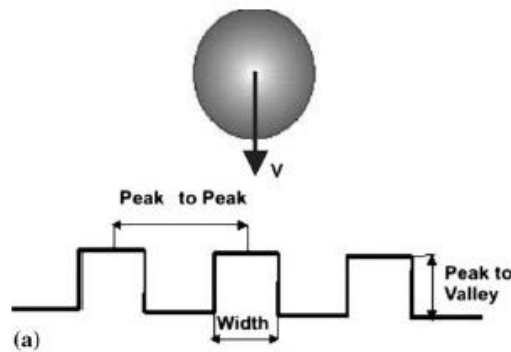


Figure 11. Schematic of the simulation capability of SimDrop for droplet impact on a textured surface [88]

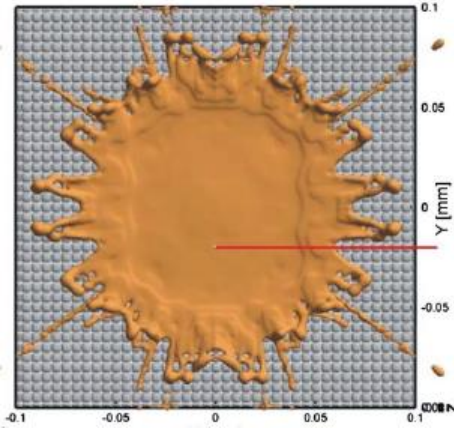


Figure 12. A splat generated after simulated droplet impact on a textured surface using SimDrop [85]

3 Experimental Results and Discussion

3.1 Grit Blasting of Reaction Bonded SiC

A substantial amount of the experimental work has been dedicated to grit blasting as well as plasma spray coating of rb SiC with a Si bond coating to gain a better understanding of this method of generating roughness on this material, the nature of the surface roughness created and its relation to the adhesion strength of the plasma sprayed coating. This initial work laid the foundation for and led to the realization of the need for the remainder of the work.

3.1.1 Process and Methods

Blast Equipment

Grit blasting was performed using a pressure-type system with a nozzle attached to a FANUC M-10iA 6-axis robot arm with R-30iB controller. A boron carbide nozzle with bore diameter of 6mm was used. The samples were fixed to metal structures inside a blasting cabinet using double sided tape. The nozzle, robot and setup for automated grit blasting can be seen in Fig. 13a. Nozzle angle and standoff distance relative to the substrate, as well as traverse speed and blast pressure, were all set via the controller. Grit mass flow rate was controlled via a valve which adjusts the size of the inlet opening between pressure vessel and hose. This was measured prior to blasting experiments by inserting the nozzle into an opening of a large bucket and measuring the amount of grit which filled the bucket in thirty seconds. The robot was

programmed such that the nozzle traversed completely beyond the sample with each traverse to allow the nozzle to reach the desired speed, and with a step size of 10mm to ensure full and even blasting of the surface. The time required for the nozzle to complete a full traverse was measured to verify traverse speed.

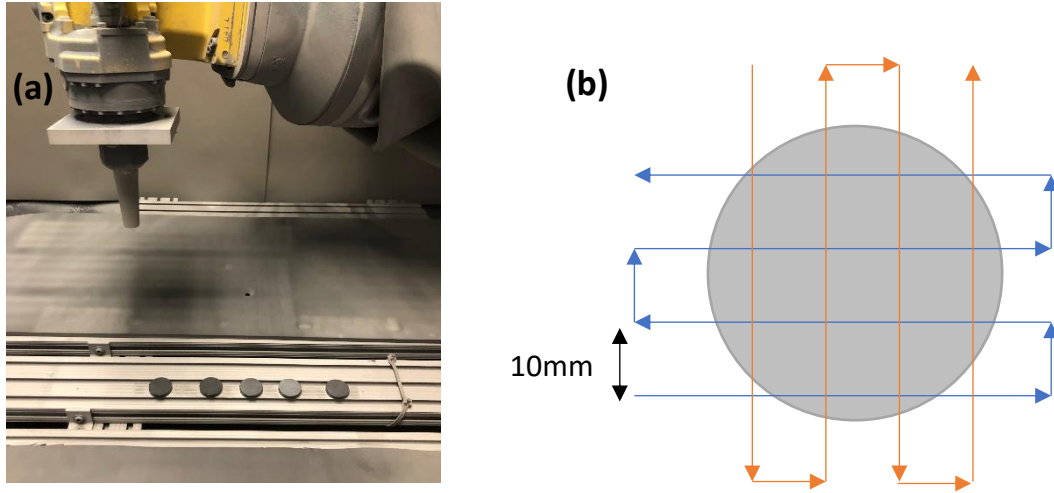


Figure 13. (a) Image of robot arm inside of blast cabinet and (b) diagram of standard grit blasting traverse pattern and step size

Substrate and Grit Material

Substrates were round, high purity, reaction bonded silicon carbide (rb SiC) buttons (M Cubed Technologies, Newark, DE, USA) with diameter of 25mm, thickness of 4.5mm, average as-received roughness (R_a) of $0.353 \pm 0.05 \mu\text{m}$ and Knoop hardness of 1080 kg/mm^2 . The buttons were cleaned with ethanol prior to grit blasting. Black 220 grit SiC ($63\mu\text{m}$) (Detroit Abrasives) was used as the blast media for all blasting trials.

3.1.2 Blast Parameter Study

Based on manual grit blasting experience, preliminary screening trials and reported studies on grit blasting of metals (8-9), a standoff distance and grit mass flow rate were seen to have little effect on induced roughness and therefore were maintained at 150mm and 0.5 kg/min, respectively, for all trials as listed in Table 1. Initially, nozzle traverse speed was varied (300, 600, 800, 1000, 1500 mm/s) at different blast pressures (50, 75 psi) to observe the effect of traverse speed on R_a (as well as other roughness parameters) and thickness loss. Results from

these initial experiments led to the use of 800 and 2000 mm/s nozzle traverse speeds for the subsequent experiments.

The repeatability of the process at baseline blasting parameters (50 psi, 85°, 800 mm/s) was evaluated by blasting three separate sets of five RB SiC buttons, measuring their average roughness using the confocal microscope method detailed above, averaging their R_a values and comparing these values for each set. The process was seen to be very repeatable as shown in Figure 14, with a very small standard deviation of 0.011 μm between the average roughness values (each bar) for the three sets. Likewise, the standard deviation for R_a within each set, shown by the error bars, was still small, but in the range of 0.07-0.1 μm . The variation within each sample was similar. In addition, the variation of the measurement method itself was observed with a standard deviation of 0.015 μm from three measurements of R_a along the same line on a sample.

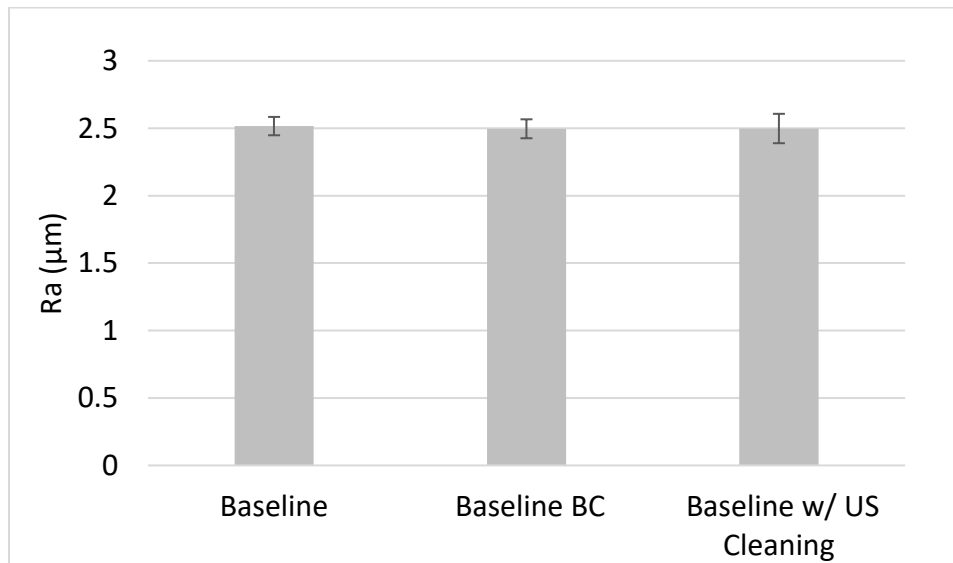


Figure 14. Plot of the average R_a for three separate sets of five rb SiC buttons blasted at baseline parameters

As shown in Figure 15, changing pressure from 25 to 50 psi at both 800 mm/s and 2000 mm/s increased average R_a by 1.27 μm . Changing pressure from 50 psi to 75 psi at both traverse speeds increased R_a by about 0.7 μm . This indicates that changes in blast pressure will increase or decrease average roughness by the same amount regardless of the nozzle traverse speed.

A very similar result was observed for the effect of changing traverse at different pressures. R_a decreased by about the same amount when the traverse speed was increased from 800 mm/s to 2000 mm/s regardless of the blast pressure used. This indicates that roughness can likely be controlled and predicted by manipulating blast pressure and traverse speed independently.

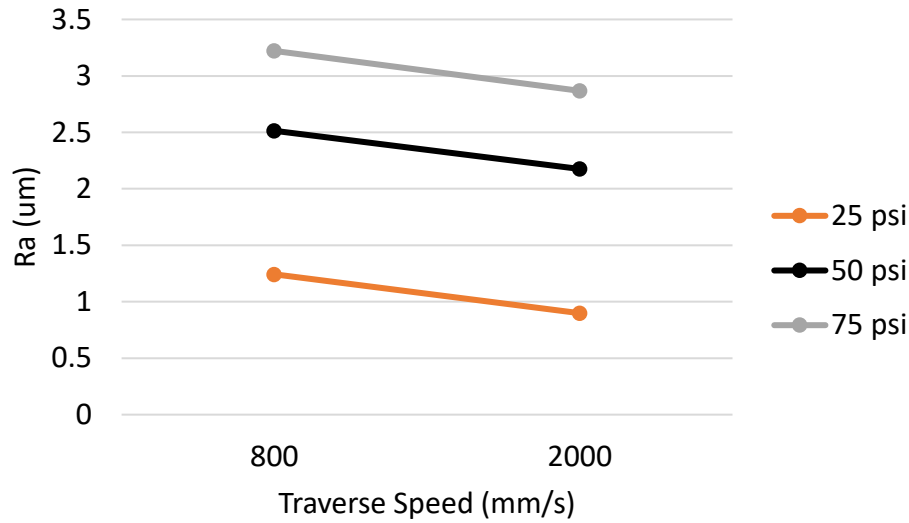


Figure 15. The effect of pressure and high traverse speeds on R_a

Blast parameters used are shown in Table 1. The bold values indicate the baseline value for that blast parameter. To study the individual effects of each parameter, the other two parameters were kept at the baseline value while the parameter of interest was varied. In addition, both traverse speeds were used at blast pressures of 25, 50 and 75 psi to further study the effects of these parameters. Five samples were grit blasted and characterized for each set of blast parameters to observe the effect on surface roughness.

Table 1. Parameters varied in grit blasting trials

Blasting Parameters	Parameter Value
Blast pressure	25, 50 , 75, 100 psi
Blast angle	45°, 65°, 85°
Traverse speed	800 , 2000 mm/s
SiC grit size	220 grit
Grit mass flow rate	0.5 kg/min
Stand-off distance	150 mm

Roughness Characterization

Characterization of the substrate surface topography before and after grit blasting was accomplished using an Olympus LEXT OLS4100 confocal laser scanning digital microscope. Two-dimensional roughness parameter values were averaged after three separate line measurements on each sample, and each line measurement consisted of five sampling lengths. Measured roughness values for initial traverse speed trials, seen in Figures 2 and 3, were from one sample. All other reported roughness values were averaged from five samples grit blasted at the same time and under the same conditions. Evaluation lengths and cutoff values used were based on the standard EN ISO 4288 [100].

Material Loss Measurement

To evaluate the material loss due to grit blasting, a Mitutoyo Digimatic MDC-1"MX micrometer with a flat, carbide tipped anvil was used to measure button thickness before and after blasting. A single measurement for sample thickness was taken to be representative of a sample set and measurements were found to be repeatable within the 1 μm resolution of the micrometer. The weight loss after grit blasting was also measured for some samples and was found to be consistent with thickness loss values. Thickness loss is used to describe the material loss in this study.

3.1.3 Results and Discussion

The Olympus OLS4100 microscope was used to obtain images of the substrate surface after grit blasting. These micrographs allowed for observation of the surface morphology in addition to the nature of the generated roughness. The typical morphology and topography of the rb SiC surface before and after grit blasting can be seen in Figure 16.

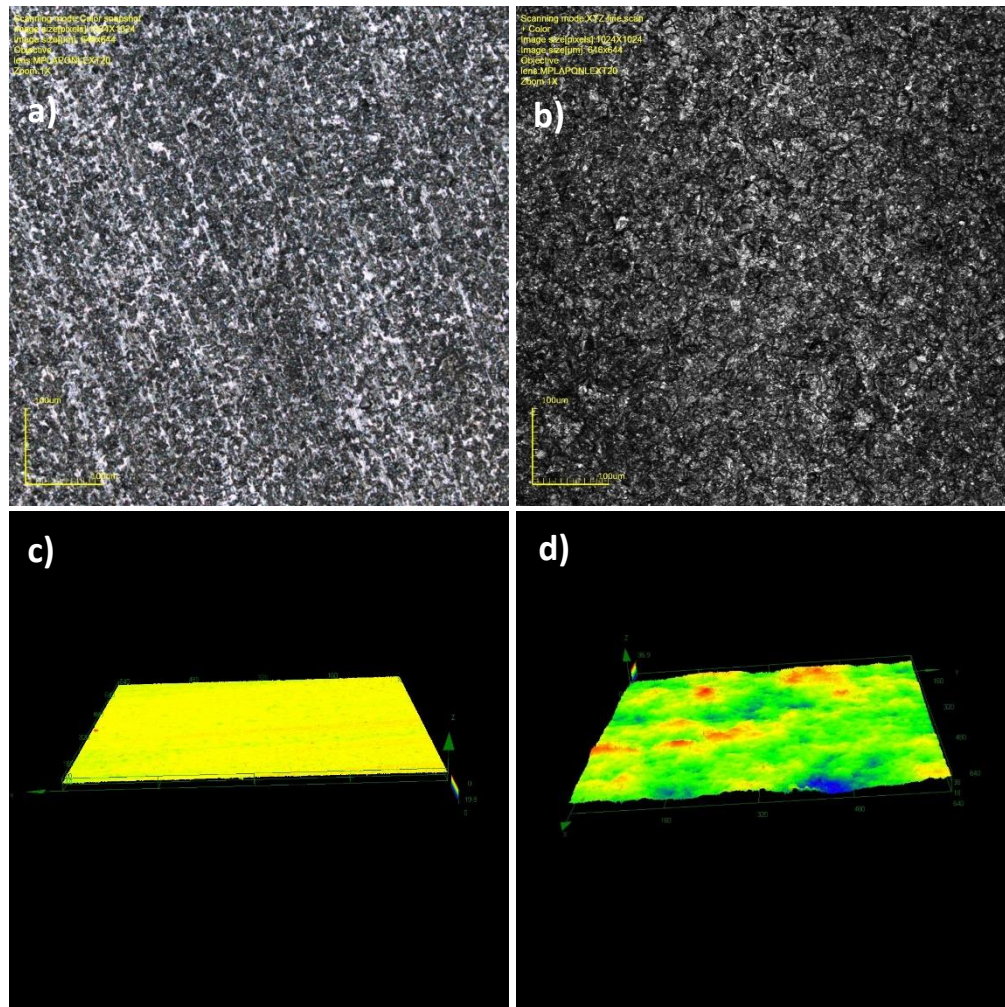


Figure 16. Optical micrographs and 3D surface maps of an a,c) as received rb SiC surface prior to grit blasting and b,d) after grit blasting

Initial trials were dedicated to studying the effect of a wide range of nozzle traverse speeds (300-1500 mm/s) on roughness and thickness loss, but also informed traverse speeds of interest for subsequent studies. Utilizing traverse speeds higher than what has previously been reported for automated grit blasting [49] showed that R_a decreased slightly with increasing traverse speed, as seen in Figure 17. The error bars correspond to $\pm$ one standard deviation. When increased from 300 to 1500 mm/s, R_a decreased by $0.7 \mu\text{m}$ at 75 psi and by $0.3 \mu\text{m}$ at 50 psi. It is also clear from these results that R_a increases with increasing blast pressure.

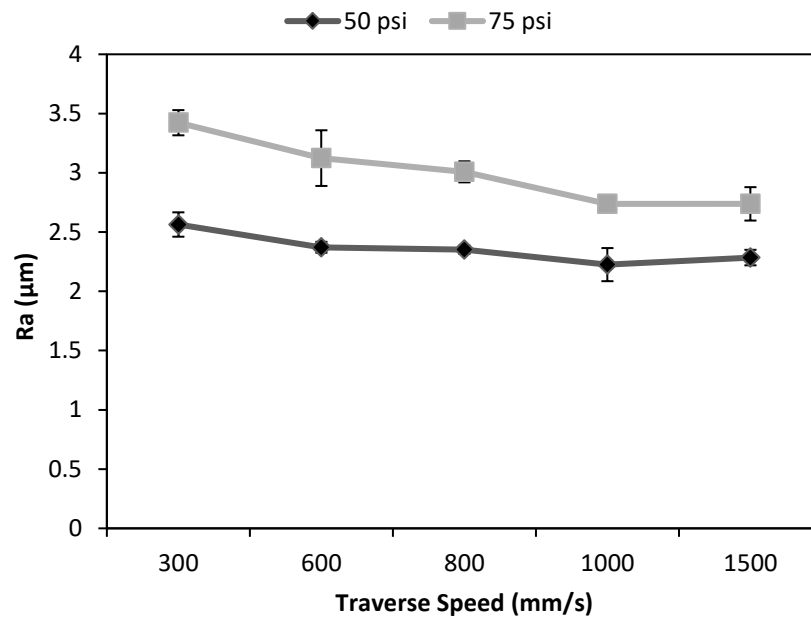


Figure 17. Plot of R_a versus nozzle traverse speed

Table 2 provides the average R_a and thickness loss for a given blast pressure and traverse speed, and Figure 18 illustrates the measured thickness loss for these trials. The change in thickness loss was much greater compared to the change in R_a as traverse speed was increased. The measured thickness loss at 300 mm/s and 75 psi was observed to be about 40 times larger than the thickness loss at 1500 mm/s. At 50 psi, the thickness loss was reduced from 21 μm to 1 μm upon increasing traverse speed from 300 to 800 mm/s. At 300 and 600 mm/s with a blast pressure of 75 psi, thickness loss was seen to be about twice that of samples when a pressure of 50 psi was used, and the higher pressure typically resulted in quite higher thickness loss. At a 1500 mm/s traverse speed, thickness loss was measured to be ≤ 1 μm (beyond the limits of the micrometer) at 50 and 75 psi. These results indicate that slow traverse speeds cause a large amount of material removal, and that material loss can be substantially reduced while maintaining high surface roughness by utilizing high traverse speeds during blasting.

Table 2. Average R_a and Thickness Loss of rb SiC after automated grit blasting with varied pressure and traverse speed

		Traverse Speed (mm/s)				
Pressure (psi)		300	600	800	1000	1500
50	Average R_a (μm)	2.56 ± 0.1	2.37 ± 0.05	2.35 ± 0.04	2.23 ± 0.14	2.28 ± 0.07
	Thickness Loss (μm)	21	8	1	1	1
75	Average R_a (μm)	3.42 ± 0.11	3.12 ± 0.23	3.01 ± 0.09	2.74 ± 0.01	2.74 ± 0.14
	Thickness Loss (μm)	39	14	8	3	1

Based on the results shown in Table 2, empirical relations for both thickness loss and R_a were developed as a function of nozzle traverse speed and blast pressure when the nozzle standoff distance is 150 mm, blast angle is 85° , step size is 10 mm and 220 SiC grit is used. The responses can be predicted by these equations:

$$\text{Thickness Loss } (\mu\text{m}) = 10.487 - 0.0276(\text{Traverse Speed}) + 0.26(\text{Pressure}) - 0.00055(\text{Traverse Speed} \times \text{Pressure}) + 0.000036(\text{Traverse Speed}^2)$$

$$R_a (\mu\text{m}) = 1.384 - 0.00048(\text{Traverse Speed}) + 0.026(\text{Pressure}) + 0.00000049(\text{Traverse Speed}^2) - 0.0000142(\text{Traverse Speed} \times \text{Pressure})$$

The statistical model for thickness loss has a high R^2 value of 0.98 and an adjusted R^2 value of 0.96, indicating that the model agrees well with the actual values and only 2% of the total variation is not explained by the empirical relationship based on R^2 . The p value of 0.0003 for the model of thickness loss, as well as the p values for each factor all being <0.02 , indicates the high significance of the model and the factors. The statistical model for R_a also has high R^2 and adjusted R^2 values of 0.99 and 0.98, respectively. Only 1% of the total variation in R_a is not explained by the empirical relationship based on R^2 . The agreement between the R^2 and adjusted R^2 , as well as the model p value of <0.0001 and factor/interaction p values <0.02 , indicates the significance of the model and the empirical relationship.

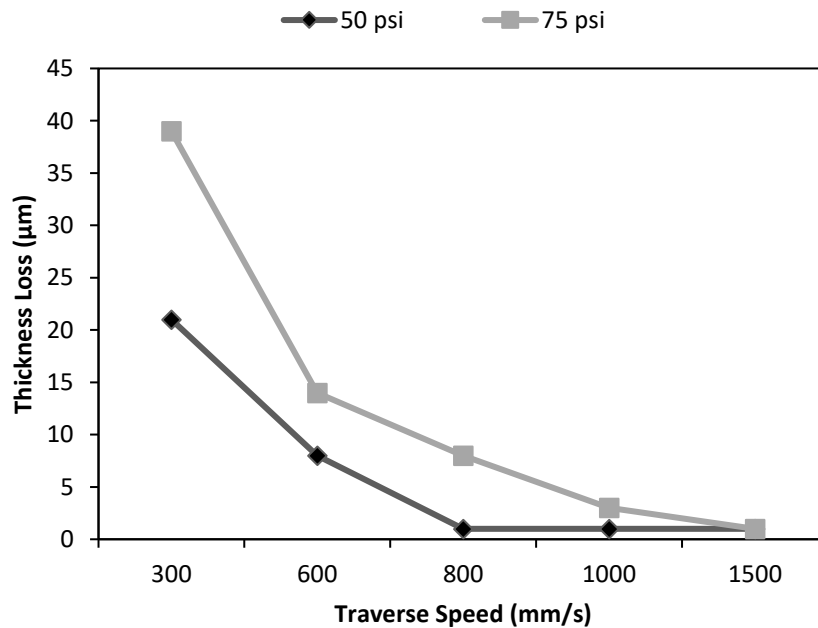


Figure 18. Plot of thickness loss versus nozzle traverse speed

The effects of blast pressure, angle and nozzle traverse speed on surface roughness are discussed below. Plotted values are averaged from five samples and error bars correspond to $\pm$ one standard deviation.

Blast Pressure

Blast pressure was found to have a strong correlation with most 2D roughness parameters of interest. R_a , R_v , R_z , R_{sm} and $R_{\Delta q}$ all increased with increasing pressure, as illustrated in Figures 19-23. Blast pressure exhibited the highest linear correlation with R_a , increasing from 1.24 μm at 25 psi to 3.76 μm at 100 psi. The linear correlation between pressure and R_v , R_z and $R_{\Delta q}$ was also relatively high, while an R^2 value of 0.66 was observed for R_{sm} . In the case of R_v , plotted in Figure 20, increasing pressure from 75 to 100 psi did not result in a further increase in the valley depth, possibly due to greater material removal and reduced valley size. R_{sm} increased substantially from $\sim 113 \mu m$ to $\sim 368 \mu m$ when pressure was increased from 25 to 50 psi but showed little change with the increase to 75 or 100 psi.

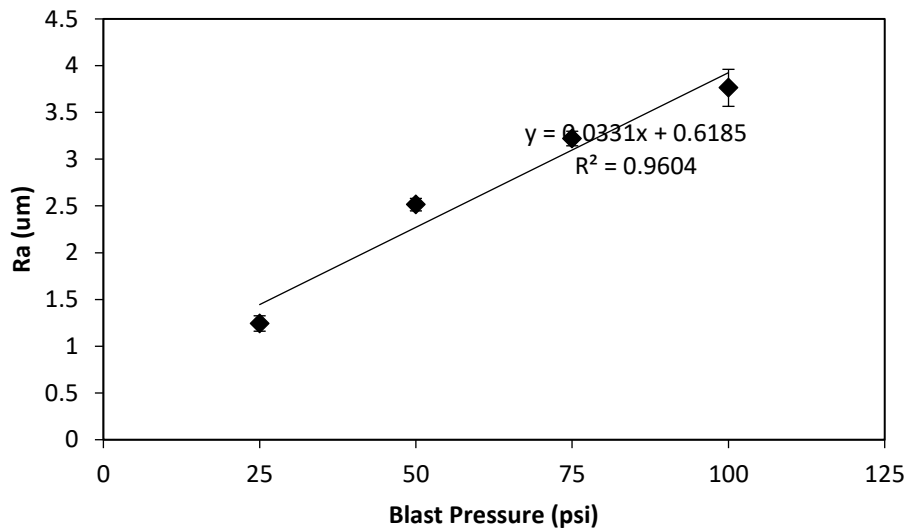


Figure 19. Plot of R_a versus blast pressure

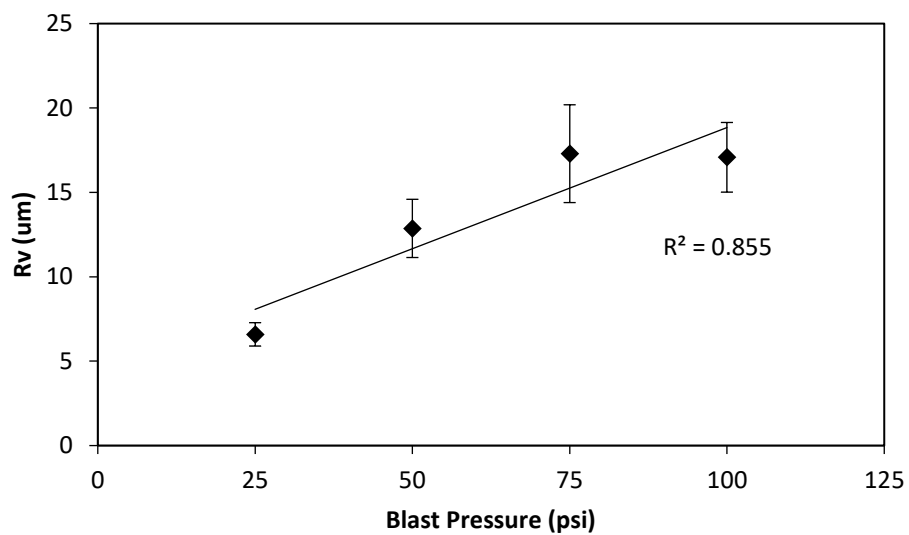


Figure 20. Plot of R_v versus blast pressure

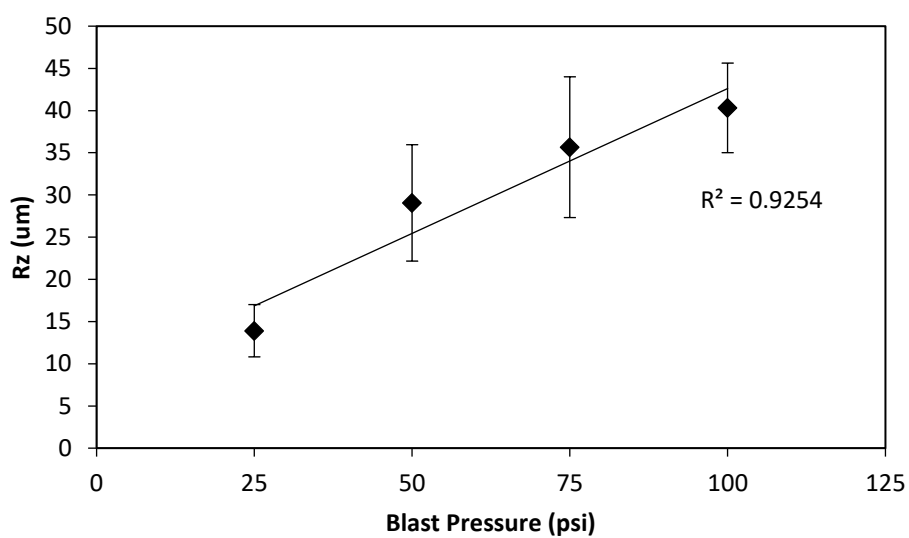


Figure 21. Plot of R_z versus blast pressure

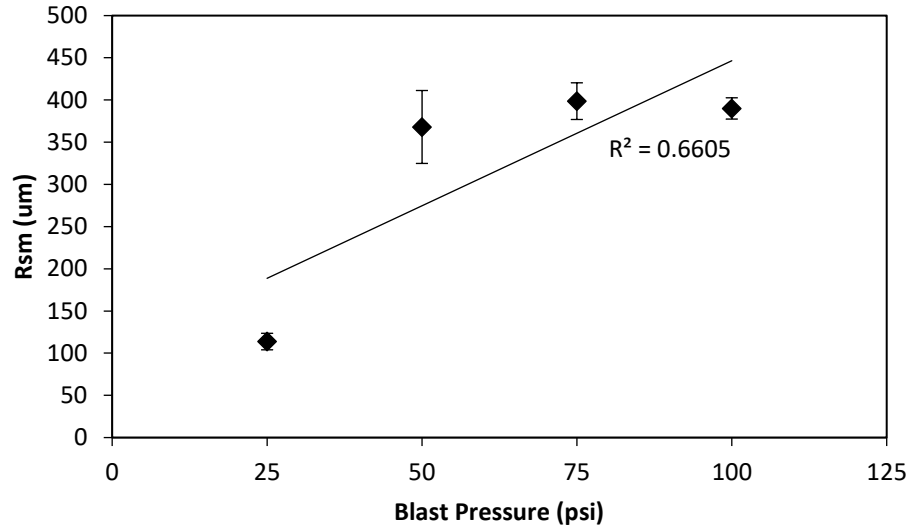


Figure 22. Plot of R_{sm} versus blast pressure

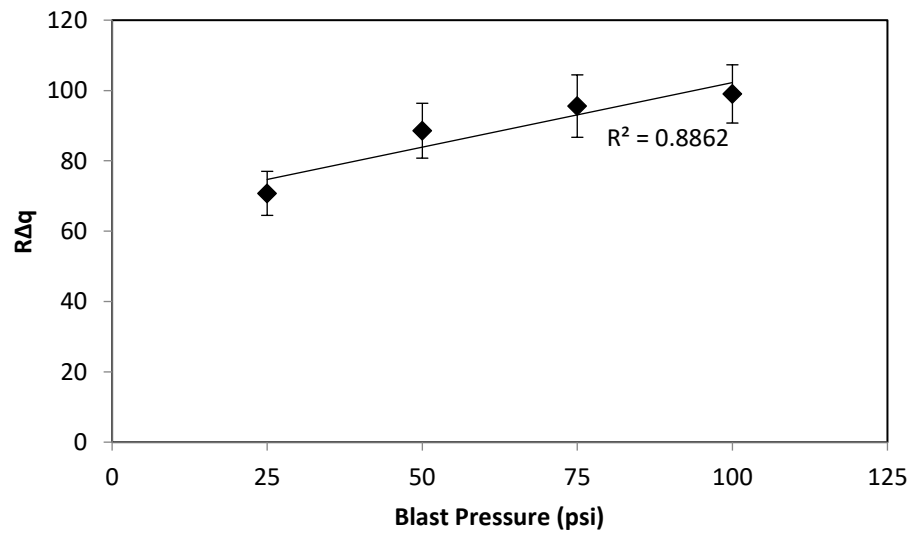


Figure 23. Plot of $R_{\Delta q}$ versus blast pressure

Blast Angle

Blast angle was observed to have a lower linear correlation with each roughness parameter of interest. The linear correlation was highest between blast angle and R_a with an R^2 value of 0.89, shown in Figure 24. Figures 24-28 illustrate that increasing blast angle from 45° to 65° resulted in an increase in each roughness parameter. For R_v , R_z , R_{sm} and $R_{\Delta q}$ (Figures 25-28), increasing angle from 65° to 85° resulted in either a small decrease or a leveling off, while R_a

showed a small increase. R_a was the only parameter which was significantly different between a 65° and 85° blast angle, $p < 0.001$, while all other roughness parameters showed no significant difference between these two angles, $p > 0.05$. For R_a , R_v and $R_{\Delta q}$, both 85° and 65° were significantly different from 45° blast angle, $p < 0.05$, but R_z was only significantly different between 65° and 45°, $p = 0.046$. R_{sm} was not statistically different between any of the three blast angles, $p > 0.1$. Though blast angle may be used to control roughness parameters, the relationship between angle and these parameters is not necessarily linear for the range of angles tested, and with blast angles between 65° and 85°, other blast parameters must be used to control roughness.

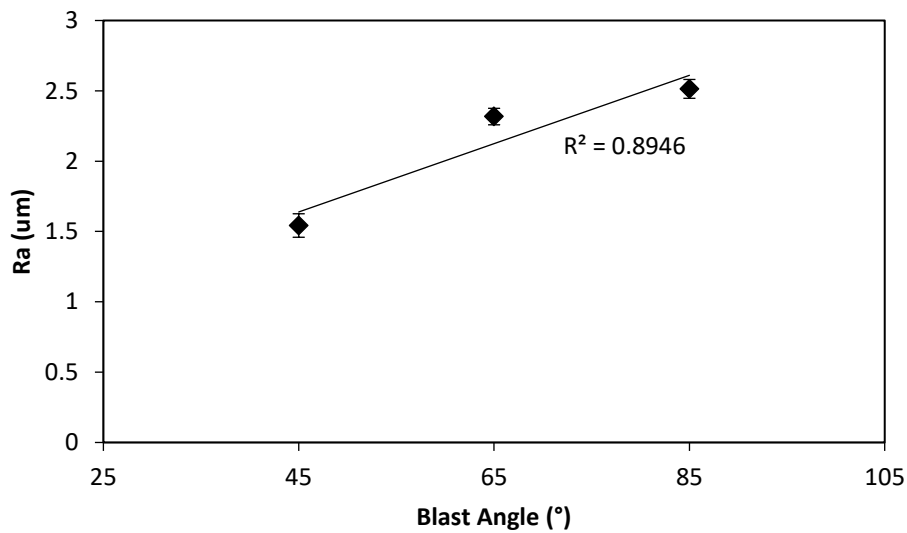


Figure 24. Plot of R_a versus blast angle

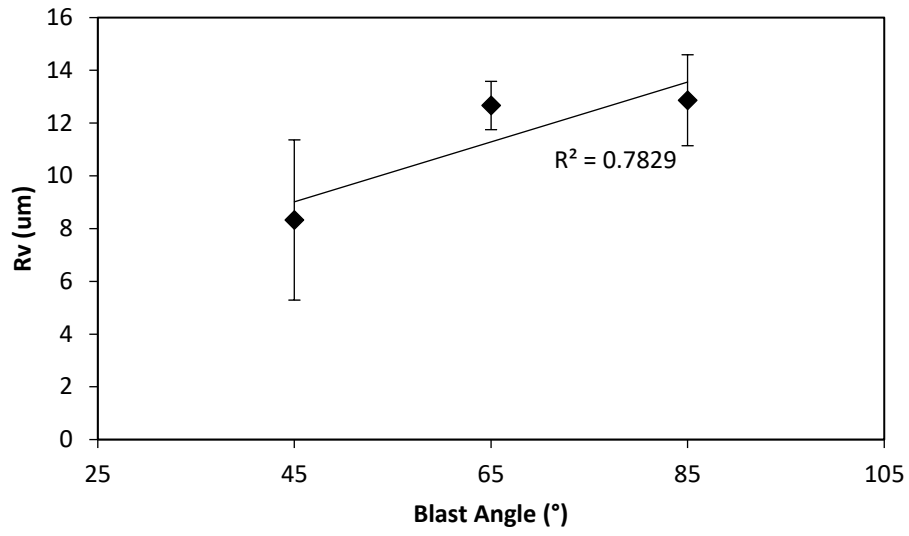


Figure 25. Plot of R_v versus blast angle

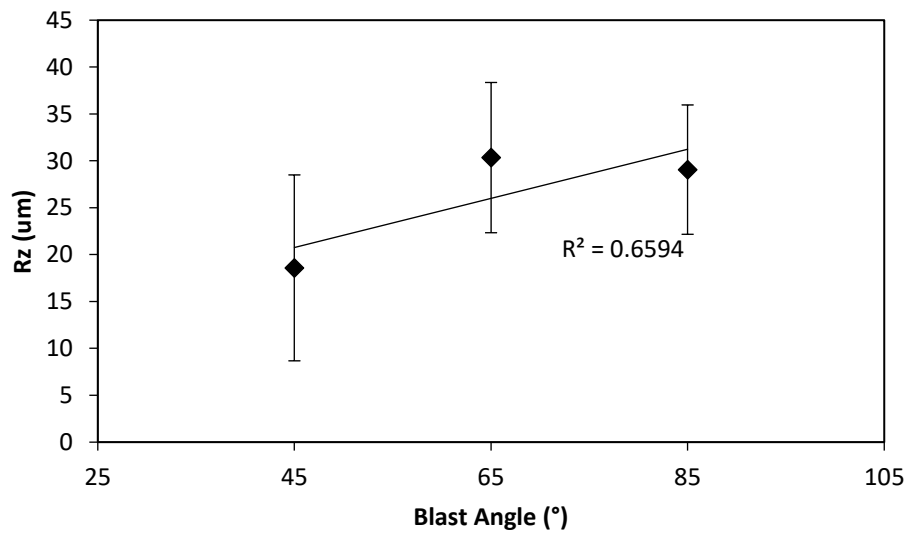


Figure 26. Plot of R_z versus blast angle

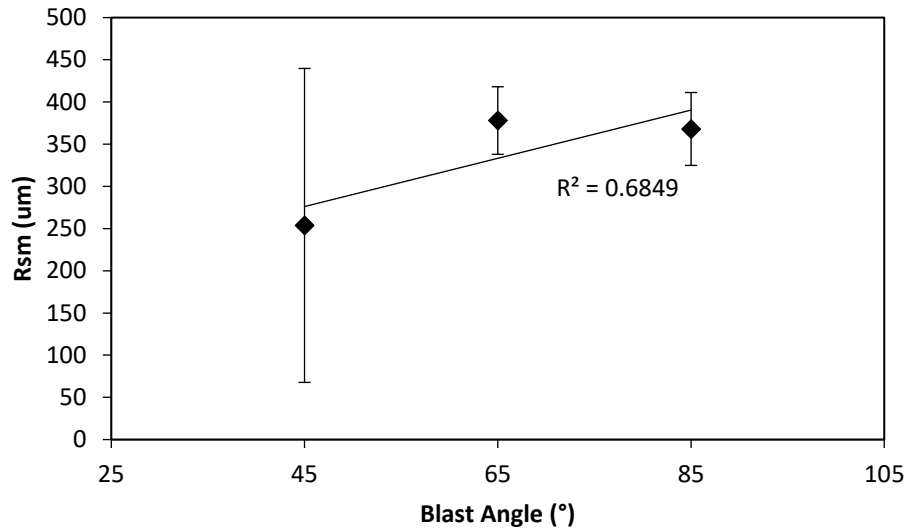


Figure 27. Plot of R_{sm} versus blast angle

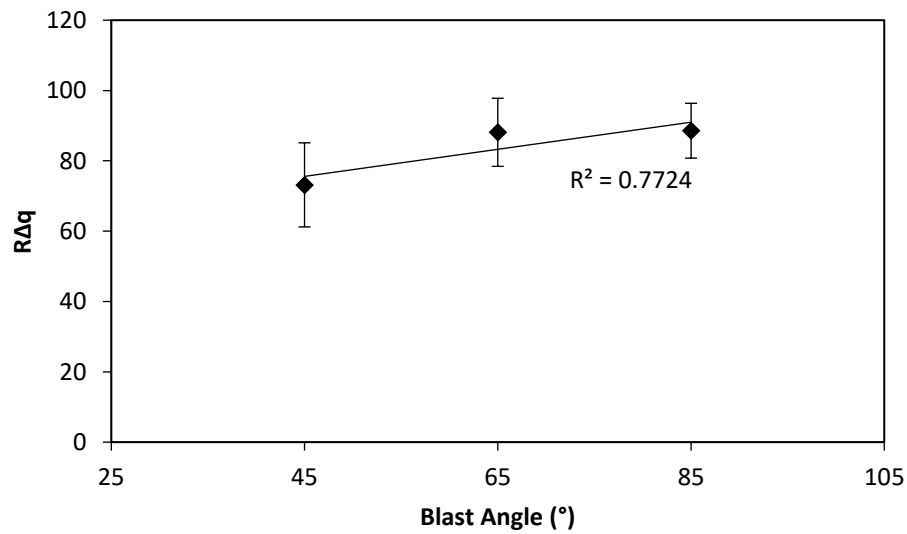


Figure 28. Plot of $R_{\Delta q}$ versus blast angle

Traverse Speed

High nozzle traverse speeds of 800 and 2000 mm/s were used to more closely study the achievable roughness at these speeds while minimizing thickness loss. Increasing blast nozzle traverse speed from 800 to 2000 mm/s resulted in a small decrease in the mean value of each roughness parameter of interest, however the difference was only statistically significant for R_a ,

$p < 0.05$ (Figures 29-33). Compared to blast pressure and angle, nozzle traverse speed has a much smaller effect on induced roughness.

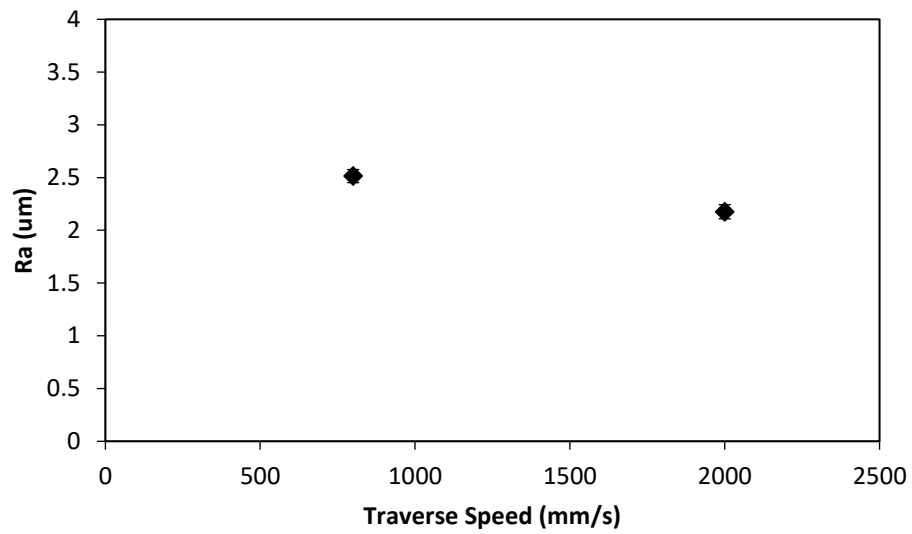


Figure 29. Plot of R_a versus nozzle traverse speed

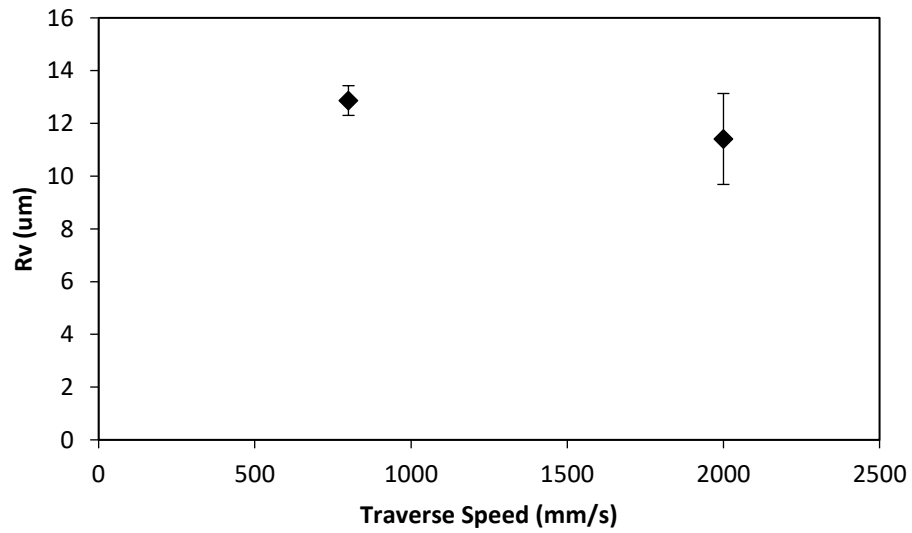


Figure 30. Plot of R_v versus nozzle traverse speed

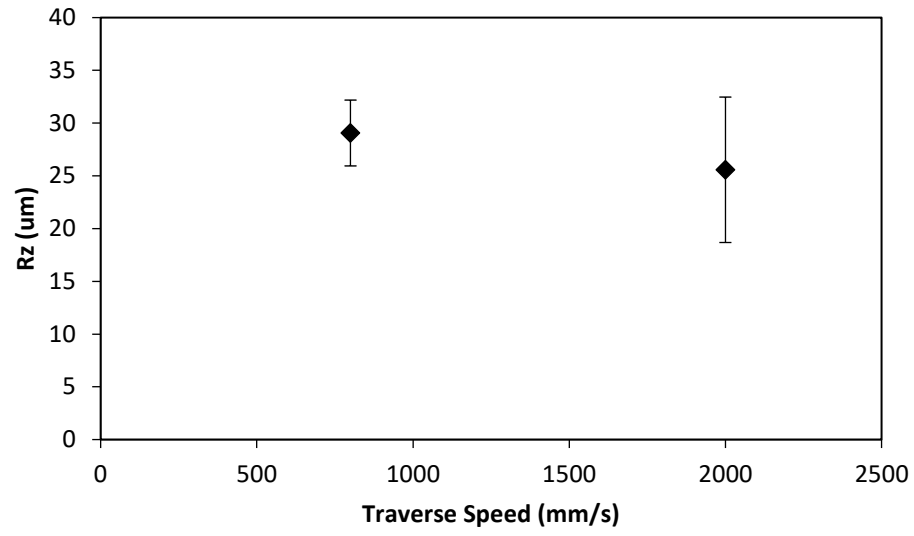


Figure 31. Plot of R_z versus nozzle traverse speed

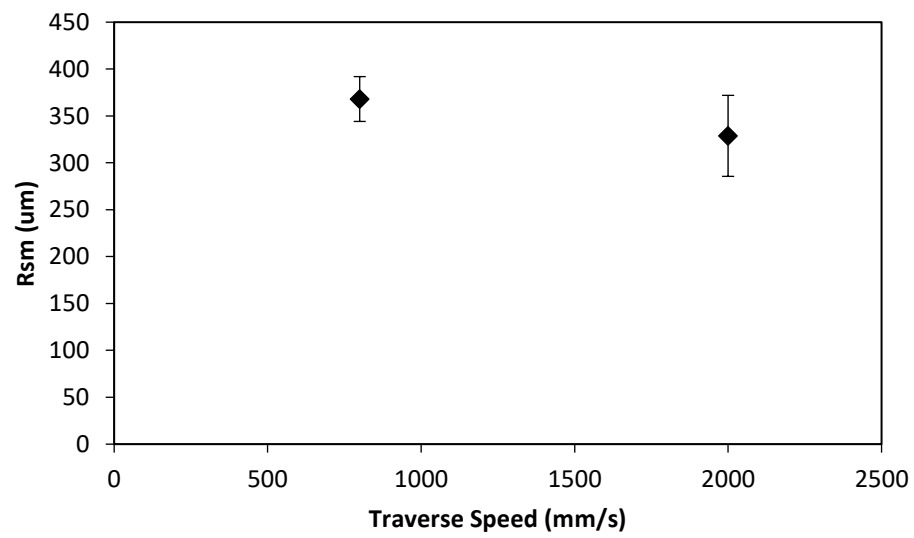


Figure 32. Plot of R_{sm} versus nozzle traverse speed

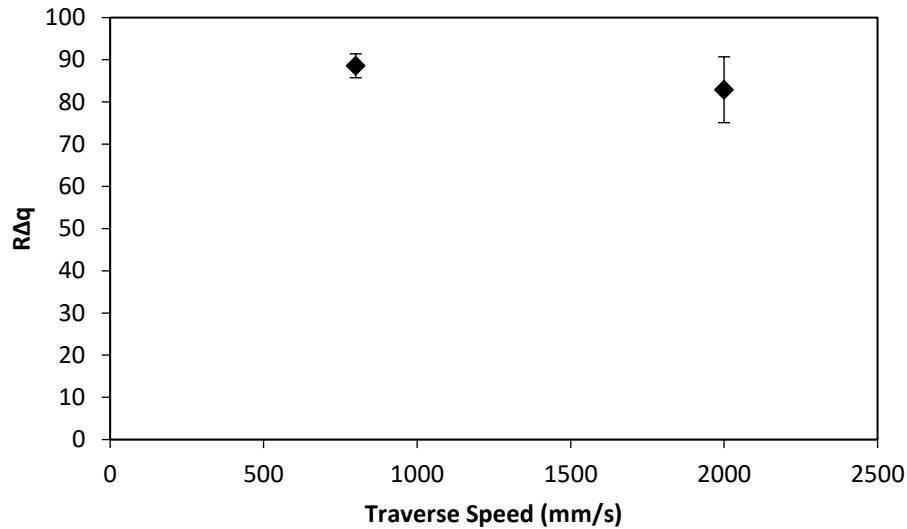


Figure 33. Plot of $R_{\Delta q}$ versus nozzle traverse speed

Despite the small effect of traverse speed on surface roughness, it was observed once again to play a dramatic role in the thickness loss after grit blasting, even when comparing two relatively high traverse speeds, 800 and 2000 mm/s. Figure 34a shows the measured thickness loss values after grit blasting with various pressures and traverse speeds. The decrease in thickness loss upon increasing traverse speed to 2000 mm/s from 800 mm/s was substantial, especially for blast pressures of 50 and 75 psi. Thickness loss was shown to be about 5 times higher at 800 mm/s for both 50 and 75 psi. In Fig. 34, thickness loss and R_a values are included for an rb SiC sample which was manually grit blasted at 50 psi, an approximately 70° blast angle, 100 mm/s traverse speed and six inch standoff distance in two orthogonal passes with the same grit media for comparison with those blasted in this study using an automated setup. From Figure 34a, it is clear that the high traverse speed capability of automated blasting greatly reduces the measured thickness loss compared to that of manual grit blasting and slower traverse speeds. In addition, induced R_a values are similar or higher than that of the manually blasted sample despite the greatly reduced thickness loss. Comparing the standard deviations of average R_a for automated and manual grit blasting, it can be seen that they are similar, however the standard deviation remains consistent for automated blasting across multiple runs with varied parameters, which isn't expected for manual blasting. The advantage of utilizing high nozzle traverse speeds was clearly seen as an average R_a of $2.87 \mu\text{m}$ was obtained with a measured

thickness loss less than 5 μm when blasted at 75 psi, 85° and 2000 mm/s, while standard manual grit blasting resulted in an average R_a of 2.21 μm and a thickness loss of 17 μm , as seen in Figure 34. Values for samples blasted at 100 psi, 800 mm/s and with the standard manual blasting procedure are included in Figure 34 for comparison, however, no samples were blasted at 100 psi and 2000 mm/s or manually at 2000 mm/s, and these values are not plotted.

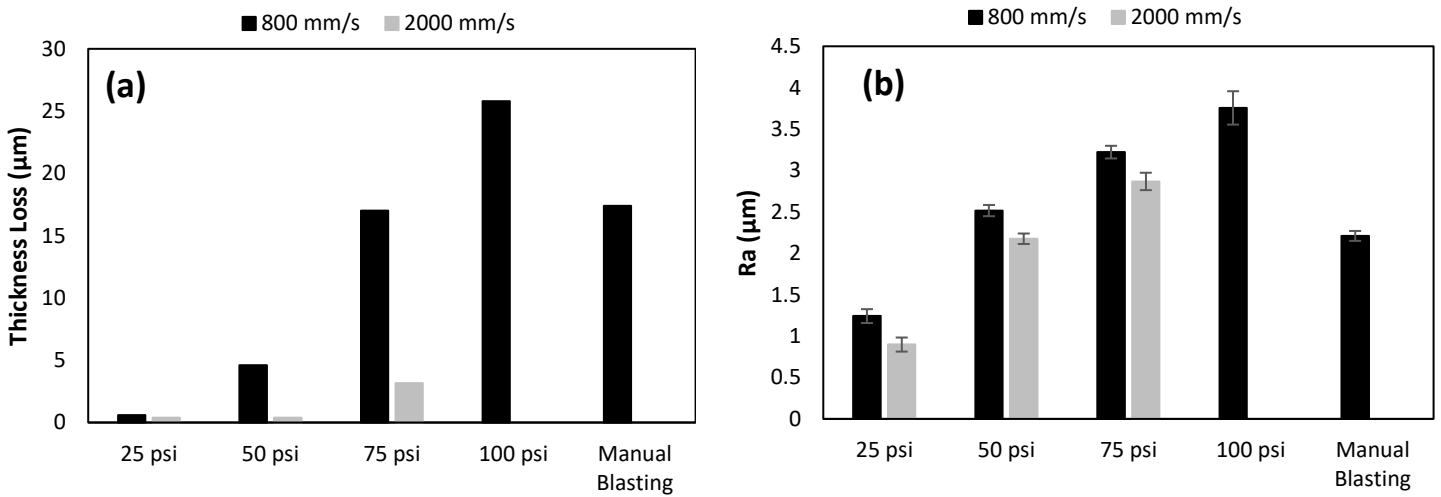


Figure 34. Plot of (a) measured thickness loss values and (b) average R_a values for samples grit blasted at different blast pressures and traverse speeds

3.1.4 Role of Substrate Material in Induced Roughness and Thickness Loss

The effects of differences in substrate material and substrate reinforcement particle size, which can be clearly observed in the Figure 35, on the degree of induced roughness R_a and thickness loss from grit blasting were also studied. The major difference in induced roughness between materials isn't clear when higher blast nozzle traverse speeds are used but is very evident upon slowing down traverse speed during blasting, as seen in Figure 36. All samples were subject to the same grit blasting process and blasted at the same time, with blast pressure kept at 50 psi, blast angle at 85° and the nozzle traverse speed varied. At 800 and 400 mm/s, the induced roughness remained similar for all materials. Upon decreasing the traverse speed to 100 mm/s, a major difference in the induced roughness of the three harder materials with less free silicon and larger reinforcement particles, 702, 903 and BC-751, was observed. These materials

had significantly higher induced Ra values compared to the SSC-FG substrate at lower traverse speeds, despite the SSC-FG having a higher induced Ra than the other three samples when traverse speed was 800 mm/s. Interestingly, when comparing the amount of thickness loss observed for these samples under the same grit blasting parameters of 50 psi blast pressure, 85° blast angle and 100 mm/s traverse rate in Figure 37, it was significantly lower for the three substrates with the higher induced roughness. It is likely that the greater amount and size of SiC or B₄C in the samples resulted in less removal of these materials and mostly removal of Si near the surface, leading to a rougher final surface but little change in thickness. The SSC-FG still experienced substantial removal of SiC and the removal of both Si and SiC resulted in the observed greater overall thickness loss and the lower roughness. This makes clear that the induced roughness and material removal depends greatly on the material and the nature of the surface to be grit blasted. The traverse speed used during grit blasting must also be selected based on the substrate material, desired roughness and tolerable material removal.

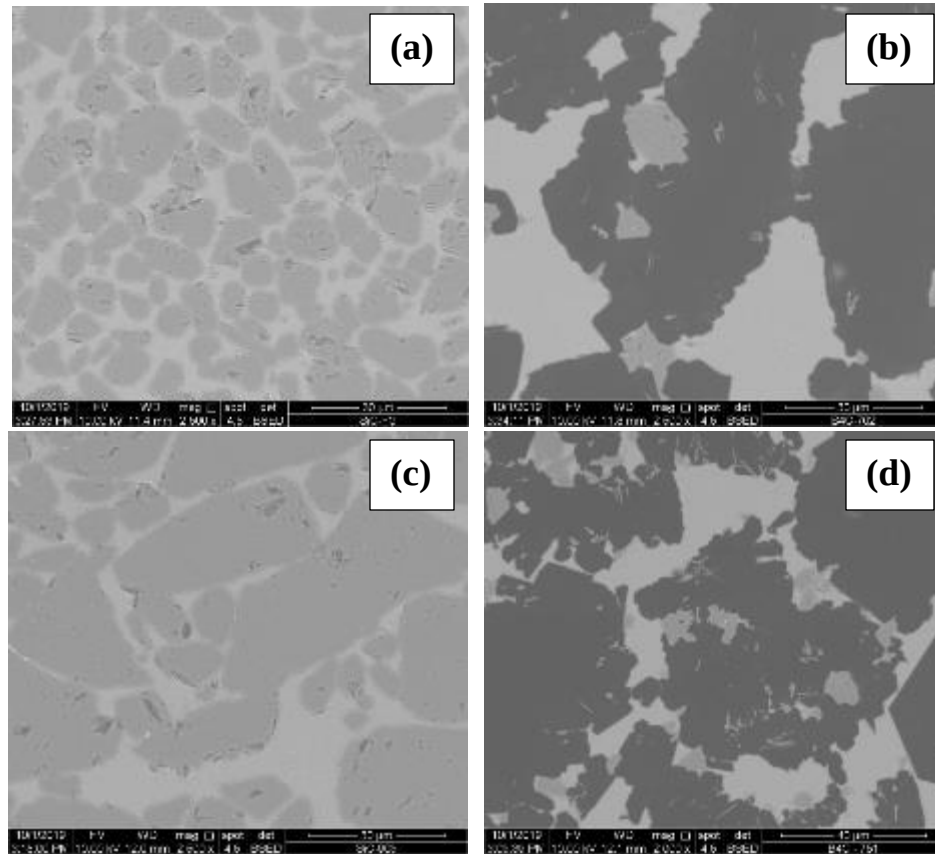


Figure 35. SEM micrographs showing the microstructure of a) SSC-FG, reaction bonded silicon carbide with 30% free Si, b) 702, reaction bonded boron carbide, c) SSC-903, reaction bonded silicon carbide with 20% free Si, d) BC-751, reaction bonded boron carbide

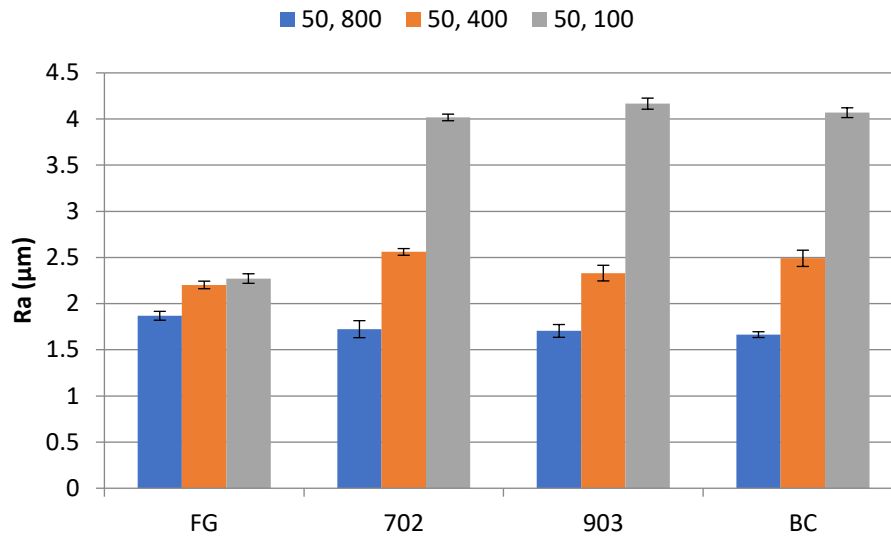


Figure 36. Plot of average R_a induced on silicon carbide (FG and 903) and boron carbide (702 and BC) substrates at an 85° blast angle, 50 psi and 800, 400 and 100 mm/s traverse speed

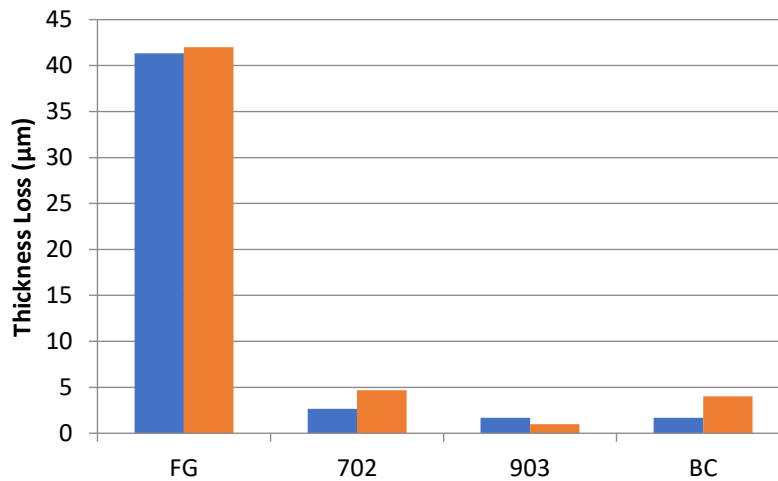


Figure 37. Plot of average R_a induced on silicon carbide (FG and 903) and boron carbide (702 and BC) substrates at an 85° blast angle, 50 psi and 100 mm/s traverse speed

3.1.5 Summary

This study observed the effect of the grit blast pressure, angle and traverse speed on various surface roughness parameters (R_a , R_v , R_z , R_{sm} and $R_{\Delta q}$), as well as the measured thickness loss after automated grit blasting of rb SiC. The effect of changing these parameters on induced roughness of the ceramic substrate was found to exhibit similar trends to what has been

reported for metal substrates. The achievable roughness for this rb SiC substrate blasted with 220 SiC grit and the range of studied blast process parameters was demonstrated. Increasing blast pressure resulted in an increase in each roughness parameter of interest and had the strongest effect on roughness. Pressure also had the highest linear correlation with roughness parameters of interest. The average peak spacing R_{sm} plateaued as blast pressure increased above 50 psi and average R_v plateaued above 75 psi. The highest average R_a of 3.76 μm was achieved at the highest pressure, 100 psi, an 85° blast angle and 800 mm/s nozzle traverse speed. Increasing blast angle from 45° to 65° resulted in a clear increase in the average of each roughness parameter, while, from 65° to 85°, average R_a only slightly increased and the average of other roughness parameters either remained the same or decreased slightly. Changes in blast angle between 65° and 85° have very little effect on surface roughness, while smaller blast angles below 65° can result in a noticeable reduction in roughness. Increasing nozzle traverse speed caused a slight decrease in the average value of each roughness parameter but resulted in a substantial reduction in thickness loss. Higher traverse speeds can be utilized to minimize material removal and thickness loss without greatly affecting roughness. Traverse speed and thickness loss have an asymptotic relationship in which the magnitude of the change in thickness loss decreases and approaches zero with increasing traverse speed. As such, changes in traverse speed at slower speeds (<600 mm/s) will have a greater effect on thickness loss than changes at higher speeds (>600 mm/s).

The effect of substrate material, amount of free Si and microstructure on grit blasting induced roughness and material/thickness loss was evaluated. Reaction bonded boron carbide (702 and BC) and silicon carbide with less free Si (903) and larger SiC particles were found to behave differently relative to the SSC-FG reaction bonded silicon carbide used in all other experiments. The 702, BC and 903 substrates had roughness values which increased substantially with reduced traverse speed but saw very little thickness loss (<5 μm), while the SSC-FG experienced very little increase in roughness with a significant amount of thickness loss (>40 μm).

3.2 Plasma Sprayed Si Coating Adhesion on Grit Blasted RB SiC

3.2.1 Process and Methods

Substrates were round, high purity, reaction bonded silicon carbide (rb SiC) buttons (M Cubed Technologies, Newark, DE, USA) with diameter of 25mm, thickness of 4.5mm, average as-received roughness (R_a) of $0.353 \pm 0.05 \mu\text{m}$ and Knoop hardness of 1080 kg/mm^2 . The buttons were cleaned with ethanol prior to grit blasting. To study the effect of surface preparation and roughness on plasma sprayed Si coating adhesion strength, rb SiC substrates were treated via polishing, automated or manual grit blasting (GB) or left in their as-received condition and only cleaned with ethanol to generate a range of surface roughness's. The treatment used for each set of five buttons is shown in Table 3. The blast nozzle, standoff distance (from nozzle opening to sample surface), blast angle (relative to sample surface) and grit media remained constant for all automated grit blasting, and all blasting parameters used, variable or not, are listed in Table 4. During blasting, the nozzle traversed well beyond the samples to ensure full coverage and that target speed was reached. Sample set 6 was grit blasted in two steps, first at 50 psi and 800 mm/s, and then again at 20 psi and 1600 mm/s. Manual grit blasting was performed using a separate machine with the same grit media, blast pressure of 50 psi and angle of approximately 70° .

Table 3. Varied surface treatment and grit blasting parameters for each sample set

Sample Set	Treatment	Blast Pressure (psi)	Traverse Speed (mm/s)	Step Size (mm)
1	Smooth Polished	-	-	-
2	As Received	-	-	-
3	GB	25	1600	10
4	GB	25	800	10
5	GB	50	15	10
6	GB	50, 20	800, 1600	10
7	GB	50	800	3
8	GB	75	800	10
9	GB	75	200	10
10	GB	50	Manual	Manual

Table 4. Automated grit blasting parameters to generate a range of surface roughness

Grit Blasting Parameter	Values
Grit media	220 silicon carbide (~63 μm)
Blast pressure	25, 50, 75 psi
Blast angle	85°
Nozzle traverse speed	200, 800, 1600 mm/s
Standoff distance	150 mm
Step size	3, 10 mm

Substrates were grit blasted approximately one week before coating. In a previous experiment, grit blasting samples a few hours before coating (Baseline BC) resulted in the same average roughness and coating adhesion strength as those blasted a week prior to coating (Baseline), seen in Figure 38. This indicates that time between blasting and coating on the order of a few hours to a week has little effect on coating adhesion strength. However, samples blasted using the same parameters and the same average roughness but cleaned via ultrasonic bath directly prior to coating (Baseline w/ US Cleaning) showed quite a significant increase in adhesion strength, indicating the effect that substrate cleanliness and contamination has on the ability for the coating to adhere to the surface.

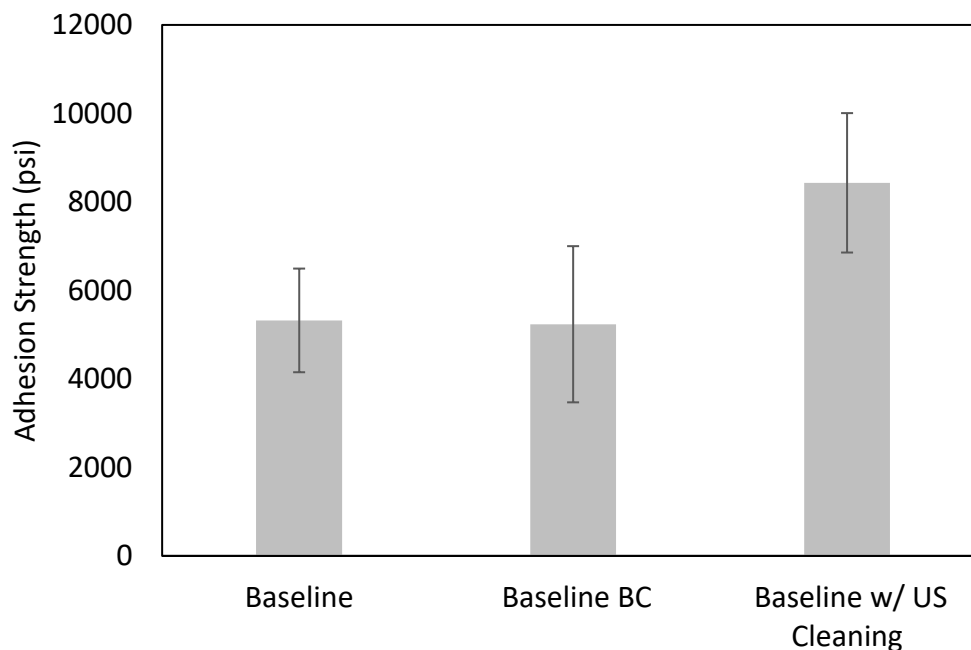


Figure 38. Plot of the average coating adhesion strength of Si on rb SiC buttons grit blasted using baseline automated blasting parameters, but with varied surface preparation before coating

To characterize the treated surfaces, an Olympus LEXT OLS4100 confocal laser scanning microscope was used to obtain 2D and 3D images of the substrate surface, measure surface roughness and 2D surface profiles. Figure 39 shows a 3D topographical map and image of a smooth polished rb SiC surface. The microstructure is seen to consist of SiC particles (dark grey) and free residual Si (light grey). Laser-scanning microscope images and 3D surface maps of the surfaces after different treatments are shown in Figure 40. R_a values were averaged after three separate line measurements on each sample, and each line measurement consisted of five sampling lengths. All reported roughness values were averaged from three out of five samples grit blasted at the same time and under the same conditions. All 3D roughness parameters were measured in three different locations on the sample surface using a 50x objective lens with a scan area of 260x260 μm and cutoff value, λ_c , of 800 μm and then averaged.

Detailed descriptions of 3D roughness parameters are provided in ISO 25178 specifications and by Deltombe et al. [89-90]. Parameters of interest are the amplitude parameters S_a , the arithmetic mean height, S_z , the maximum surface height, and S_{sk} , skewness, the functional parameter S_k , the core roughness depth, and the hybrid parameter S_{dr} , the developed interfacial area ratio. S_a provides a general measure of the average roughness of a surface similar to the commonly used parameter R_a , S_z is a measure of the distance between the highest peak and deepest valley in the measurement area, while S_{sk} describes the symmetry of a surface and gives an idea of whether the surface is dominated by peaks or valleys. S_k is a measure of the “core roughness” of a surface with the tallest peaks and deepest valleys removed. S_{dr} provides a measure of the amount of additional surface area contributed by the surface texture compared to a smooth surface.

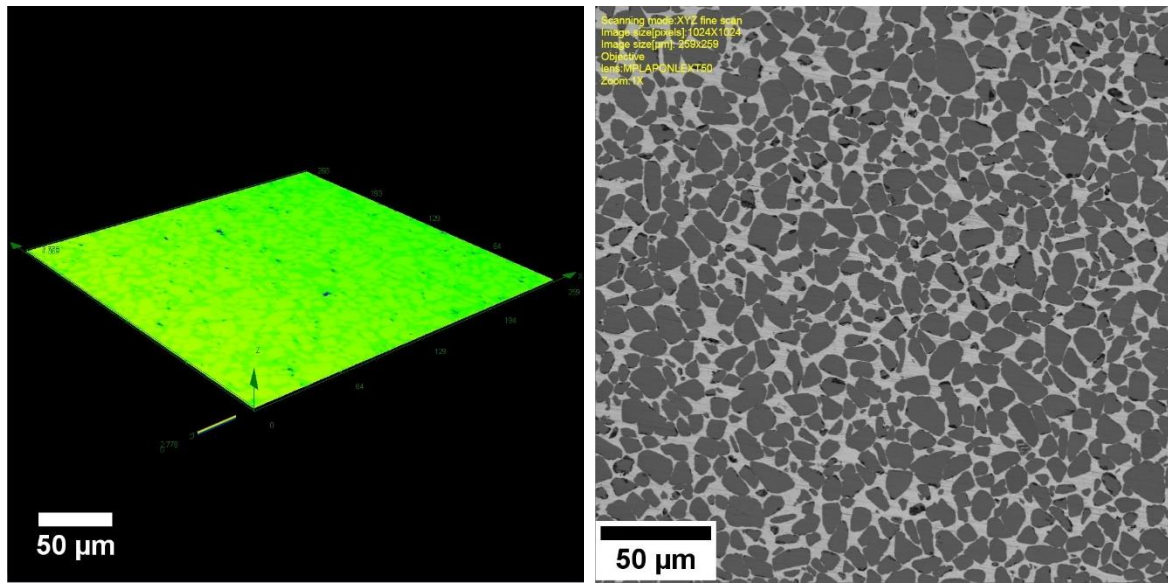


Figure 39. a) 3D surface map and b) confocal microscope image of smooth polished rb SiC surface

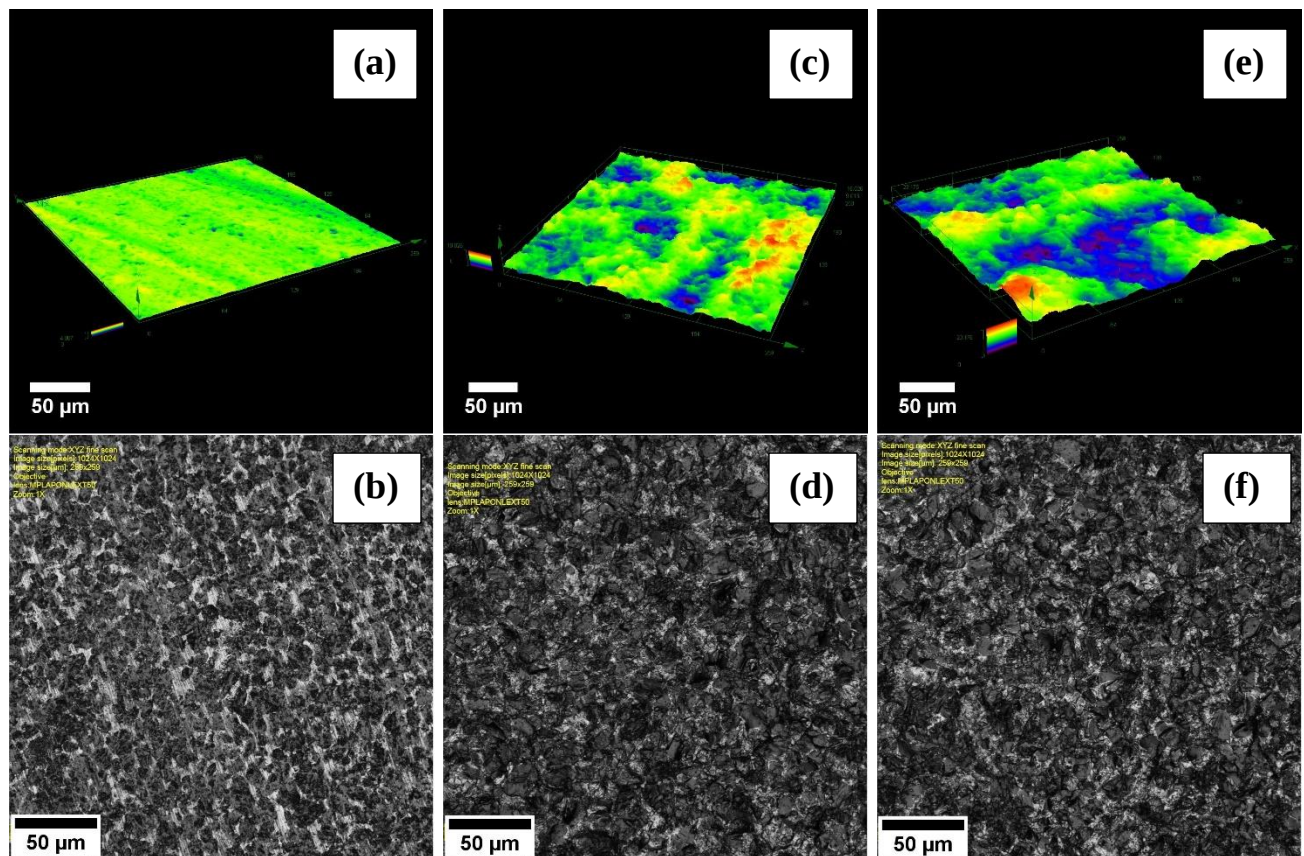


Figure 40. 3D surface maps and confocal microscope images of a,b) an as-received surface, c,d) a surface blasted at 50 psi and 800 mm/s and e,f) a surface blasted at 75 psi and 200 mm/s

The differences in the 2D surface profiles with surface treatment and grit blasting parameters can be seen in Figure 41. The y-axis minimum and maximum is 0 and 40 μm , respectively, for all profiles.

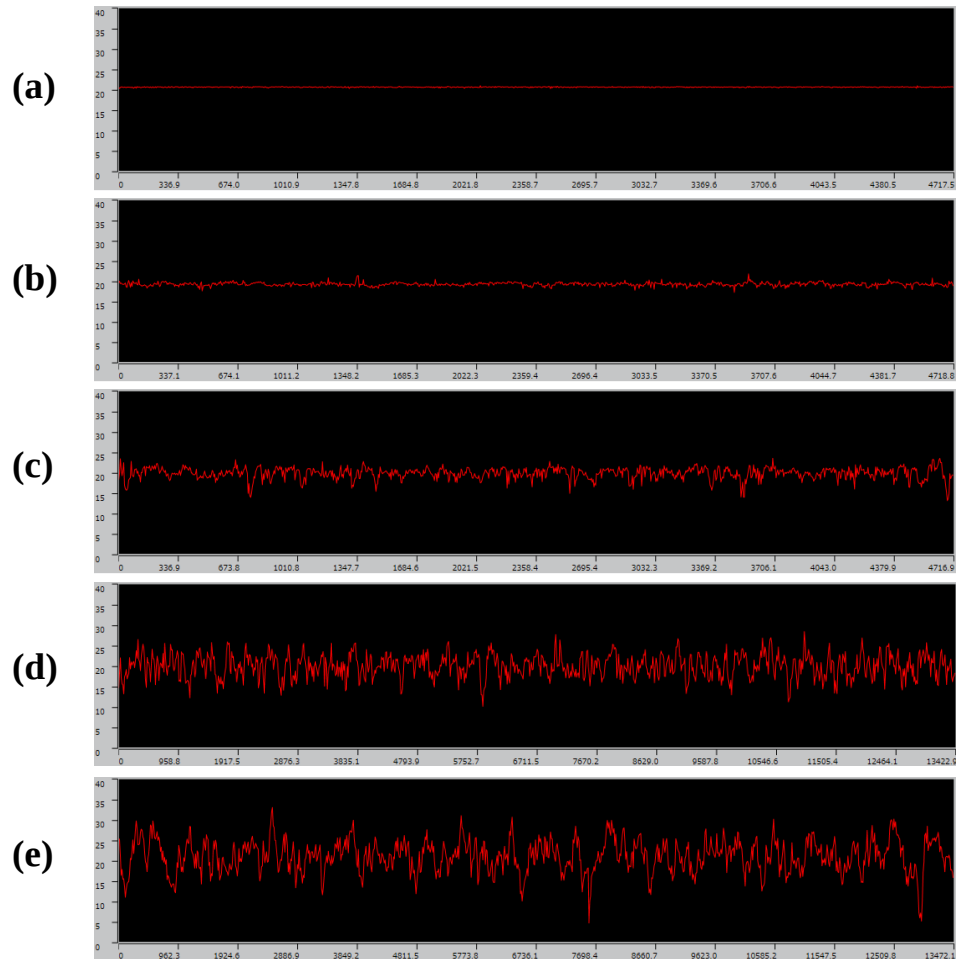


Figure 41. 2D surface profiles for rb SiC substrates after surface treatment: a) smooth polished b) as-received and wiped with ethanol, c) grit blasted at 25 psi and 800 mm/s, d) 50 psi and 800 mm/s and e) 75 psi and 200 mm/s. Y-axis goes from 0 to 40 μm for all profiles

3.2.2 Effects of Grit Blasting on 3D Surface Roughness Parameters

The effects of blast pressure, traverse speed and step size on the mean of 3D surface roughness parameters S_a , S_z , S_k , S_{dr} and S_{sk} are shown in Figures 42, 43 and 44. In each case, the blast parameter of interest was the only parameter varied while all others were held constant. Each roughness parameter increased with blast pressure, and the increase was most substantial when pressure increased from 25 to 50 psi. The difference in each parameter was less dramatic

when pressure was increased to 75 psi, and S_{sk} did not increase. Interestingly, blasting at higher pressures of 50 and 75 psi resulted in S_{sk} values closer to 0, meaning surfaces consisted of a balanced amount of peaks and valleys.

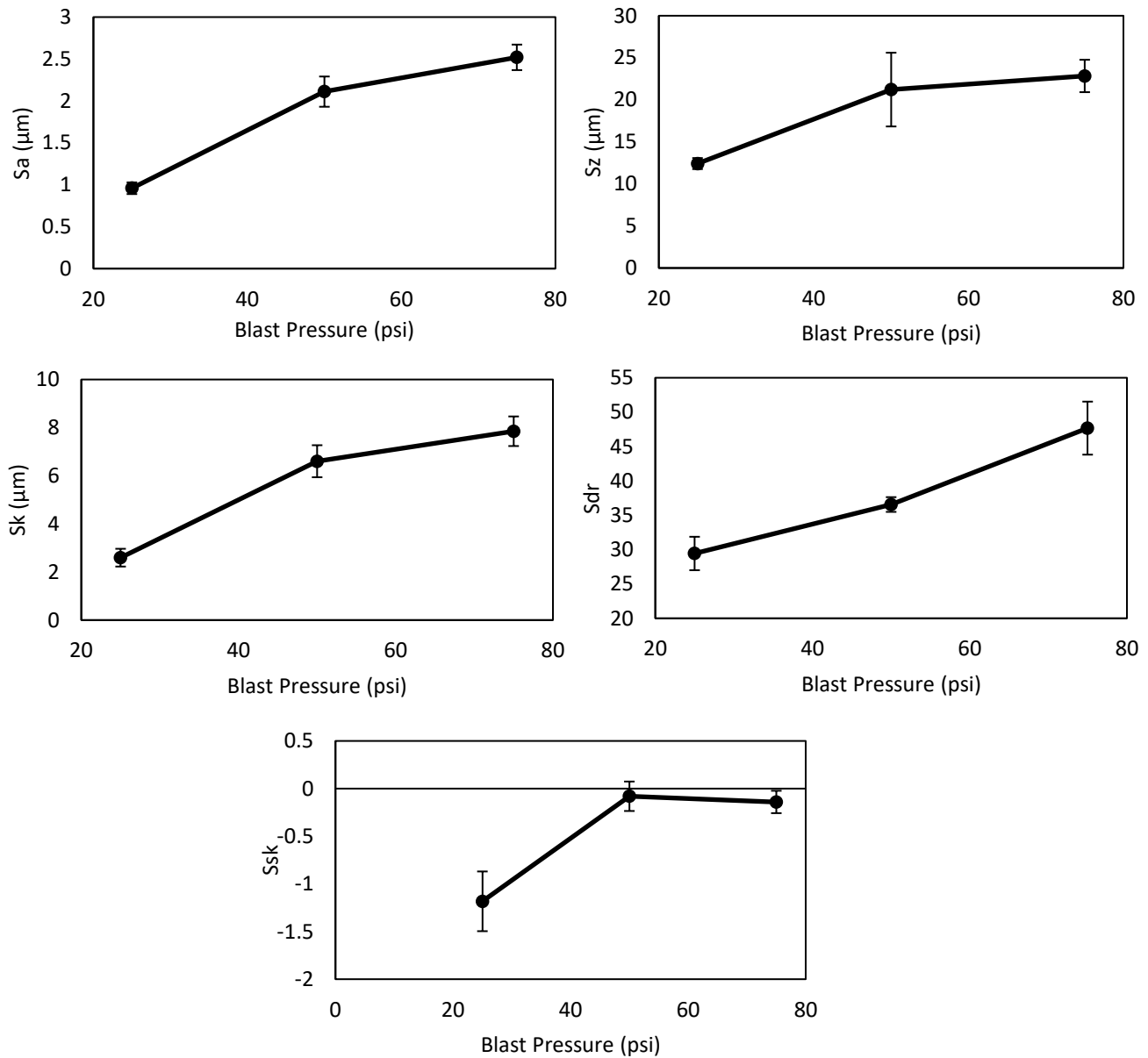


Figure 42. The effect of blast pressure on 3D roughness parameters

The effect of both blast pressure and traverse speed can be seen in Figure 43. Each parameter tends to decrease slightly with increasing traverse speed, except for Sdr when traverse speed is

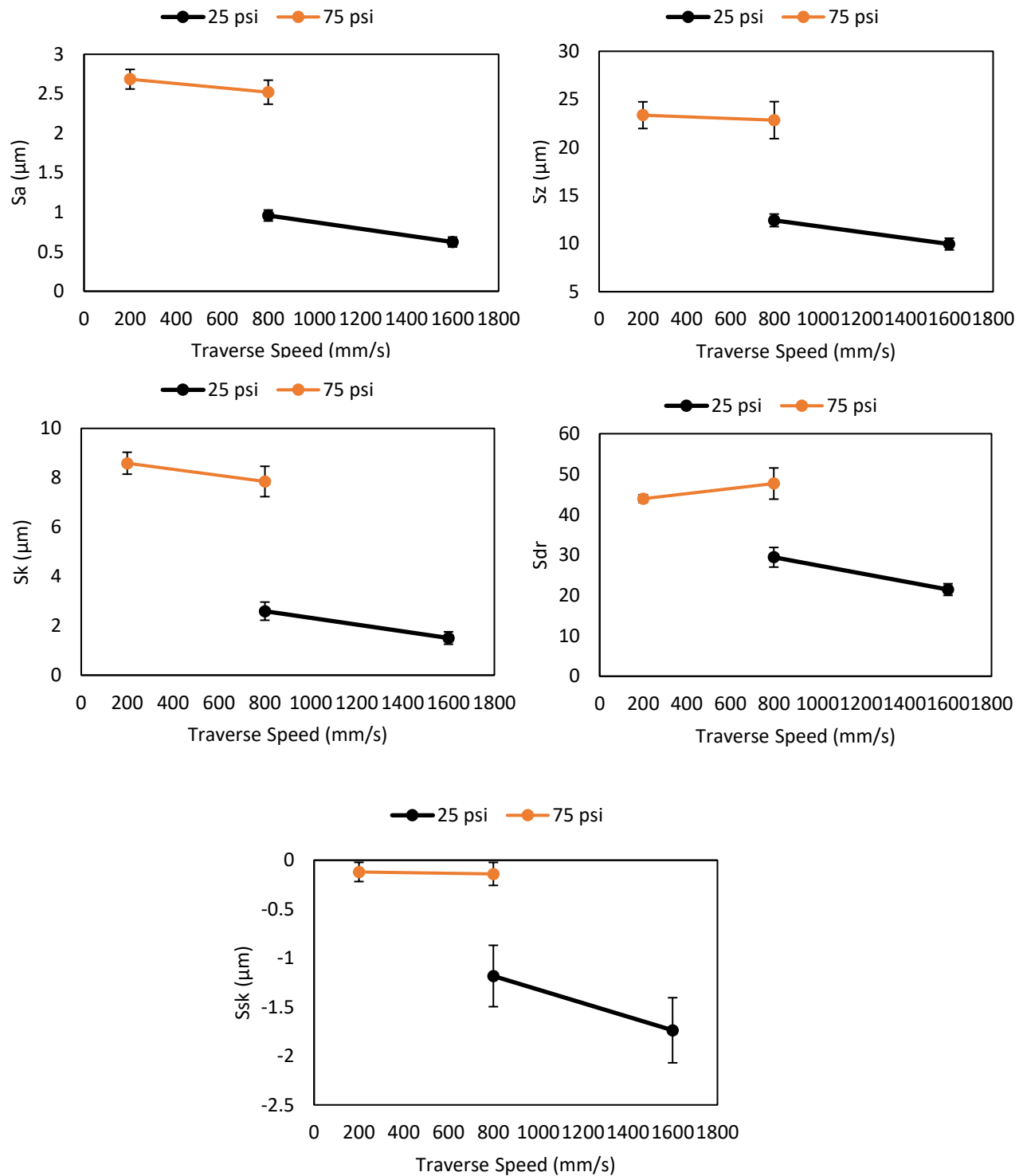


Figure 43. The effect of blast pressure nozzle traverse speed on 3D roughness parameters

increased from 200 to 800 mm/s at 75 psi. Otherwise, it is clear that increasing blast pressure from 25 to 75 psi has a significant effect on each roughness parameter. Varying step size was not seen to significantly impact roughness parameters except for Sdr as seen in Figure 44. Increasing step size from 3 to 10 mm decreased average Sdr from 42 to 36.6.

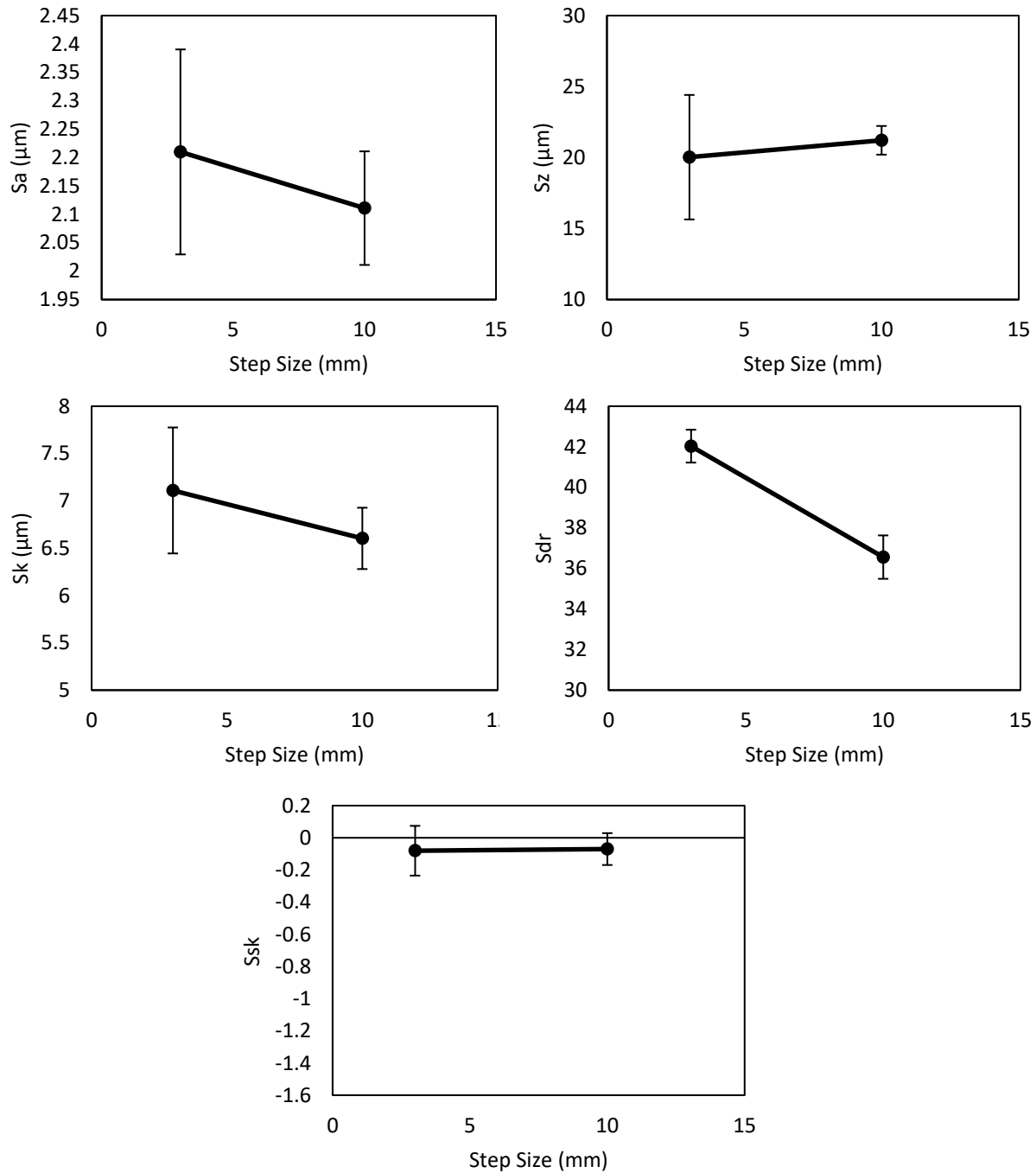


Figure 44. The effect of nozzle step size on 3D roughness parameters

Prior to depositing the Si coating via APS, samples were preheated via two traverses of the plasma torch without powder with a standoff distance of 130 mm and traverse rate of 500 mm/s. Based on previous measurements utilizing the same preheating procedure, samples were expected to be between 140 and 150 °C when coating began. The spraying powder used was pure silicon, HA 9197-2 from HAI Advanced Material Specialists, with a mean particle size of 71 μm . Five samples per surface/grit blasting treatment were preheated and coated simultaneously to keep spray parameters consistent for all samples. Substrates were coated via 11 total passes of the plasma torch to reach a coating thickness of $\sim 200\ \mu\text{m}$. A typical cross section of an rb SiC sample with a plasma sprayed silicon coating is shown in Figure 45b. Spray parameters are shown in Table 5 and the spraying setup inside the plasma spray booth at the Commonwealth Center for Advanced Manufacturing (CCAM) is shown in Figure 45a.

Table 5. Plasma spray parameters for depositing Si coating on rb SiC

Spraying Parameter	Value
Spray Gun	F4MB-XL (Oerlikon Metco)
Current, A	507
Voltage, V	74.2
Argon gas flow, NLPM	57
Hydrogen gas flow, NLPM	9
Powder gas flow, NLPM	7
Powder feed rate, g/min	29
Traverse rate, mm/s	500
Standoff distance, mm	130
Particle temperature, °C	2370
Particle velocity, m/s	344

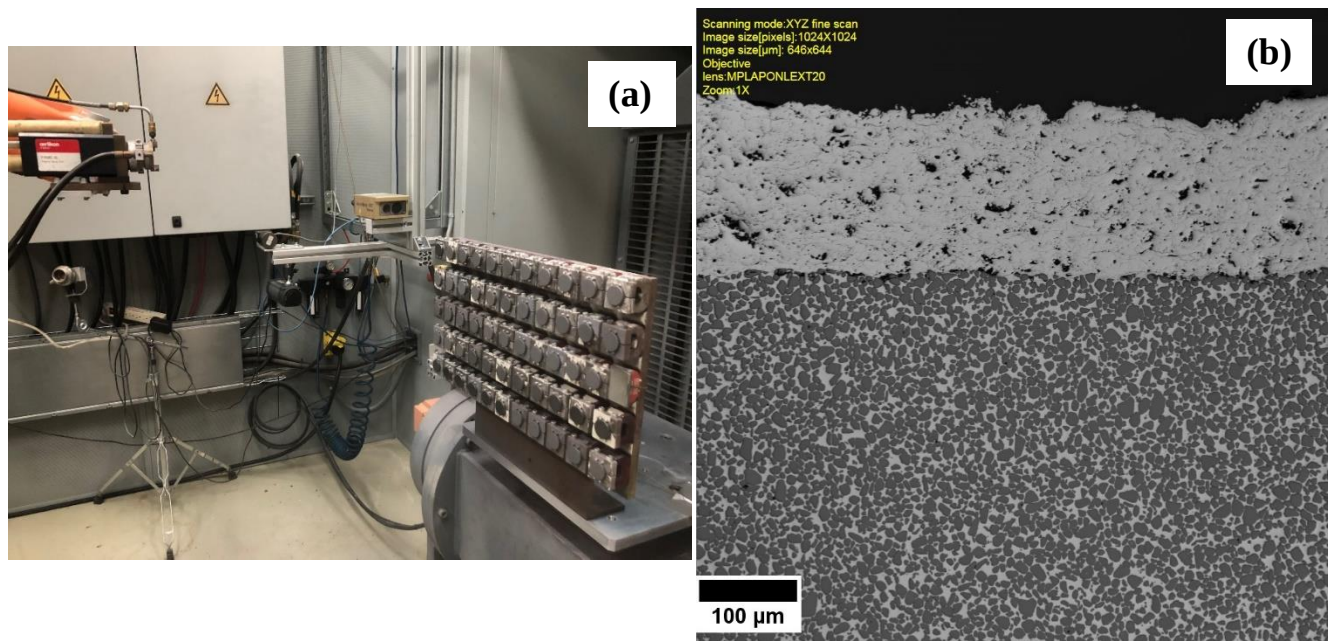


Figure 45. (a) Plasma spraying setup used to coat substrates at CCAM and (b) confocal microscope image of cross section of plasma-sprayed silicon coating on rb SiC

The adhesion strength of Si coating on rb SiC substrates was measured via tensile test according to ASTM C633 [91]. The cylindrical rb SiC buttons were joined to grit blasted tensile test bars using FM1000 epoxy, which were cured at 192 °C for 3.25 hours. The bottom, non-coated side of samples were grit blasted prior to attachment with 220 SiC grit to help ensure sufficient epoxy adhesion. Tensile adhesion tests were carried out with a head rate of 1 mm/min and five coated samples per sample set were tested and results averaged.

3.2.3 Results and Discussion

The average coating adhesion strength measured from five samples for each sample set, including samples manually grit blasted (MB) directly prior to spraying for comparison, is plotted as a function of average roughness R_a in Figure 46, with error bars representing plus or minus one standard deviation. Adhesion strength increases with increasing R_a , particularly when R_a increases above 1 μm and then seems to plateau when R_a reaches about 1.4 μm and average adhesion strength is just below 7000 psi, indicating that the adhesion strength may not be increased by further increasing R_a beyond about 1.4 μm. Increasing average roughness beyond the range observed in this study will help answer this question. However, it is important to note

that the highest average R_a was achieved using a much slower traverse speed during grit blasting compared to the rest of the samples. It is possible that this slow traverse rate, while increasing the average roughness, affects the surface in a way that is detrimental to the adhesion of the sprayed coating. When considering the results without that data point, the data shows a strong positive linear correlation between R_a and adhesion strength.

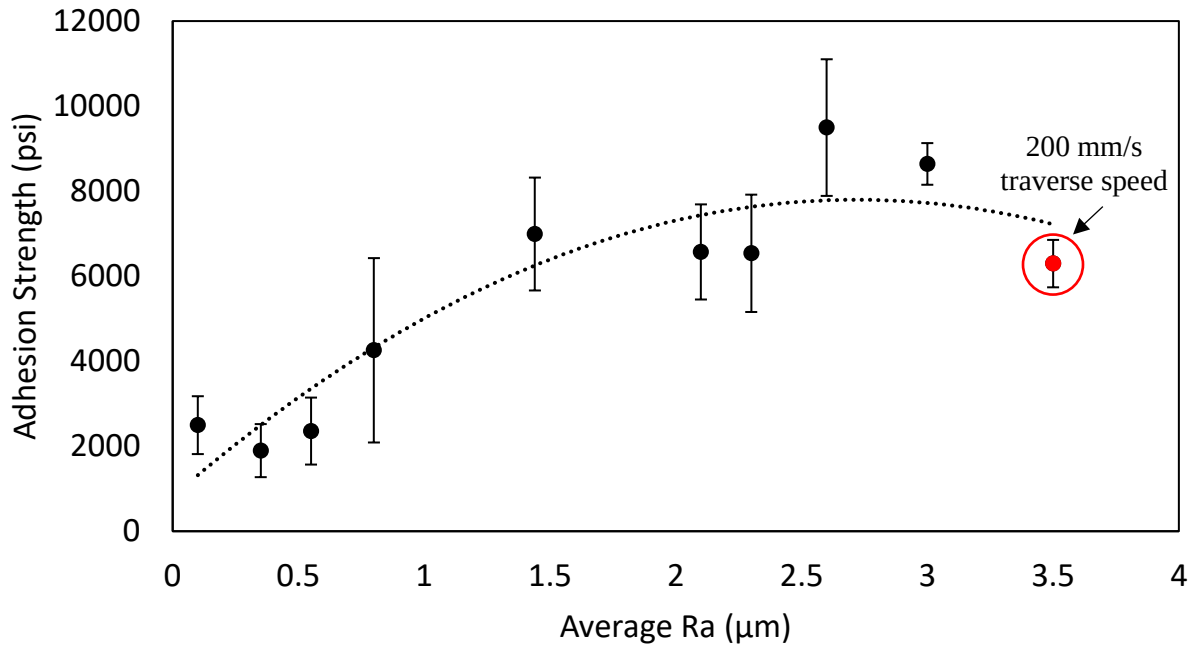


Figure 46. Si coating adhesion strength as a function of average R_a

S_a is seen to have a very similar relationship with adhesion strength as R_a , as shown in Figure 47. This makes sense as S_a provides a similar measure of roughness, just over an area instead of a line profile, but it is valuable to know these two parameters show the same relation with coating adhesion strength. S_z , plotted in Figure 48, also shows a similar increasing relationship with adhesion strength, indicating that generating taller peaks and deeper valleys in a

surface via grit blasting results in stronger mechanical interlocking between coating and substrate, though a plateauing tendency is also seen once S_z reaches a value of about 14 μm .

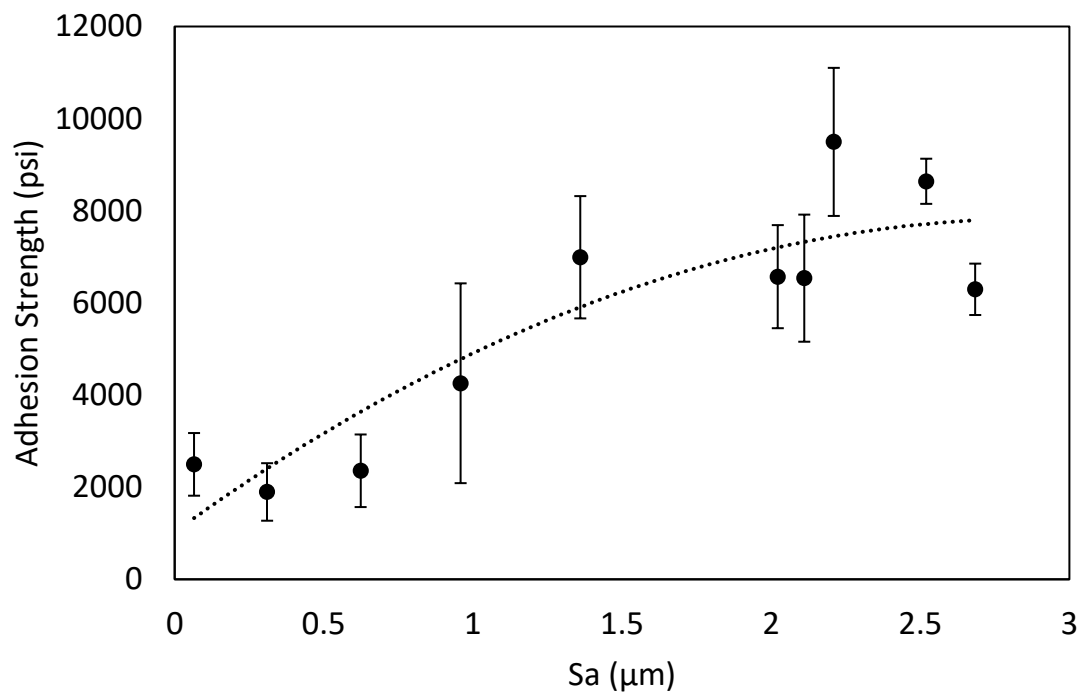


Figure 47. Si coating adhesion strength as a function of average S_z

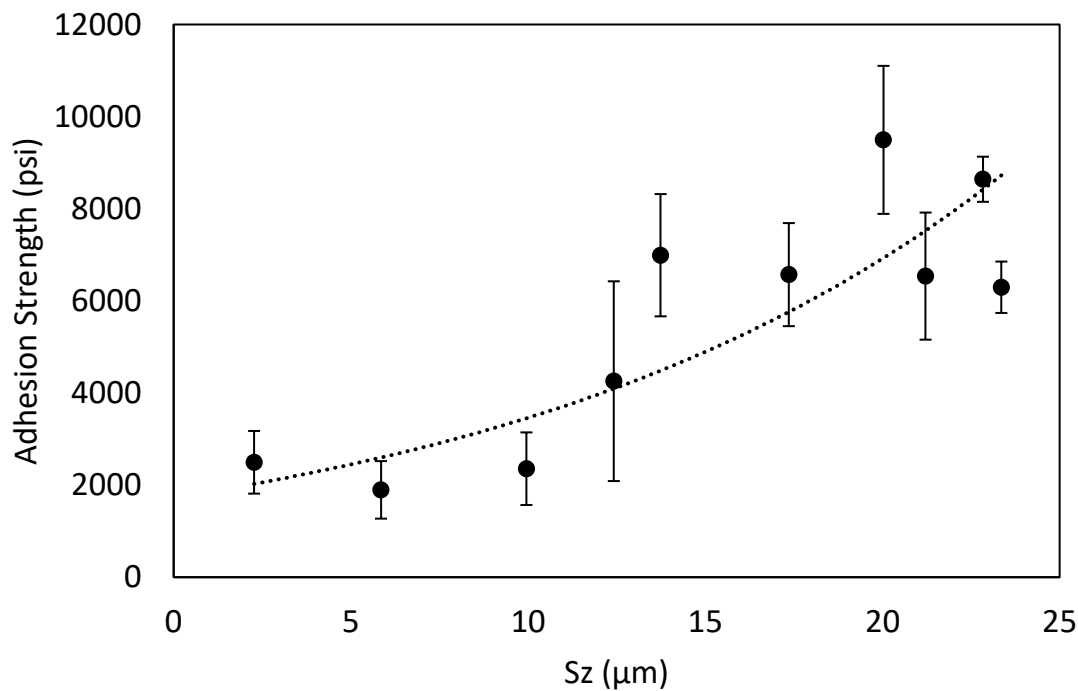


Figure 48. Si coating adhesion strength as a function of average S_z

Adhesion strength increases with increasing core roughness S_k and interfacial area ratio S_{dr} as shown in Figures 49 and 50. Similar to other measured roughness parameters, S_k was maximized when the substrate was grit blasted at 75 psi and a very slow traverse speed of 200 mm/s, however a drop in adhesion strength was observed. Without this data point, adhesion strength shows a strong, positive linear relationship with S_k . The substrates grit blasted at a traverse speed of 200 mm/s did show a change in trend with S_{dr} , shown in Figure 50, as average S_{dr} was reduced for these substrates indicating less additional surface area created by the texture. This could explain the decrease in adhesion strength observed for these substrates despite other roughness parameters being maximized, and S_{dr} is likely a strong indicator of plasma-sprayed silicon coating adhesion on grit blasted rb SiC substrates.

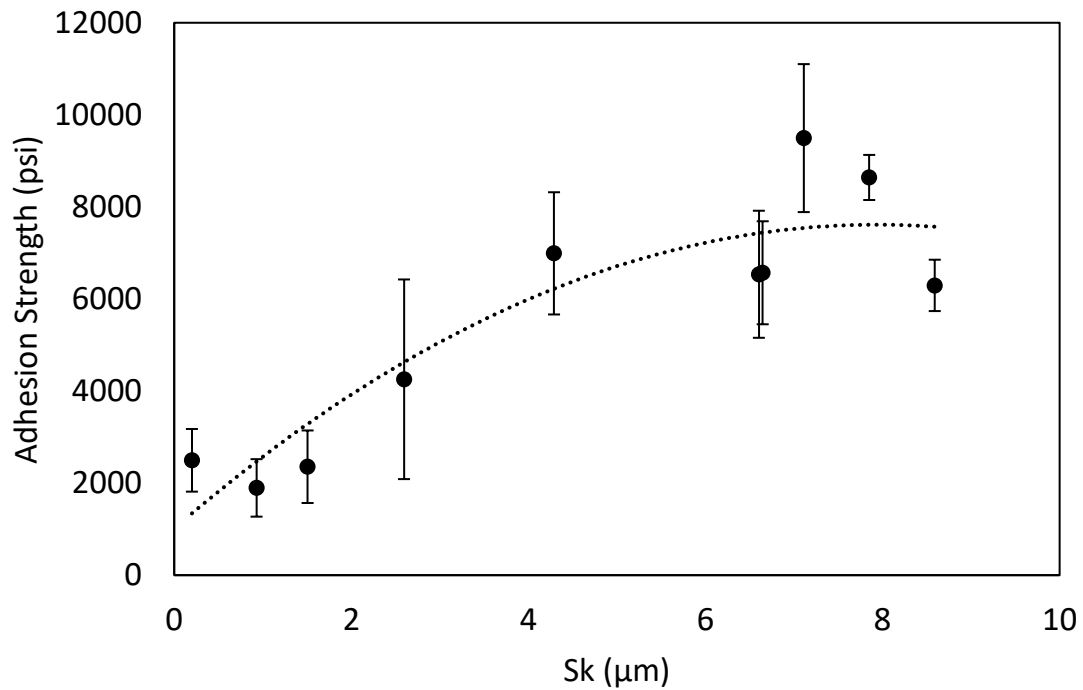


Figure 49. Si coating adhesion strength as a function of average S_k

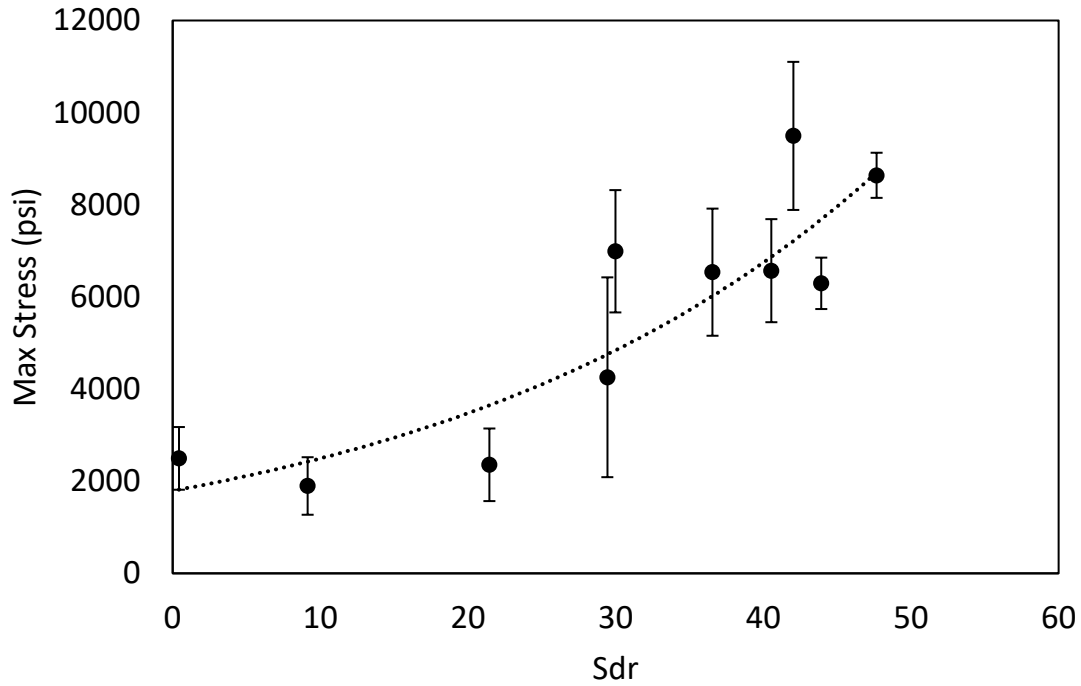


Figure 50. Si coating adhesion strength as a function of average S_{dr}

Figure 51 shows adhesion strength as a function of the surface skewness, S_{sk} . The data points in red represent substrates which were not treated with grit blasting prior to coating, but rather were either left in their as-received state after machining or smooth polished. For the grit blasted samples, adhesion strength tended to increase as S_{sk} increased. In this case, an increasing S_{sk} corresponds with the surface becoming more symmetrical as it approaches zero, at which point neither peaks nor valleys dominate the surface. All surfaces had a negative skewness value indicating more valleys than peaks on the surface, but coating adhesion strength increased and was maximized for S_{sk} values closest to zero. Figure 52 shows the relationship between S_{sk} and adhesion strength for the same grit blasted substrates as well as laser textured substrates. The laser textured substrates tended to have positive S_{sk} values indicating a greater amount of surface peaks and valleys. A similar trend to the grit blasted surfaces was observed, however, as the average coating adhesion strength increased as the skewness approached zero and adhesion was maximized on the surface with the S_{sk} value closest to zero for the laser textured surfaces. These results indicate that a symmetrical surface with a balance of peaks and valleys, given by an S_{sk} value close to zero, is ideal for optimizing plasma-sprayed coating adhesion strength on grit blasted and laser textured surfaces.

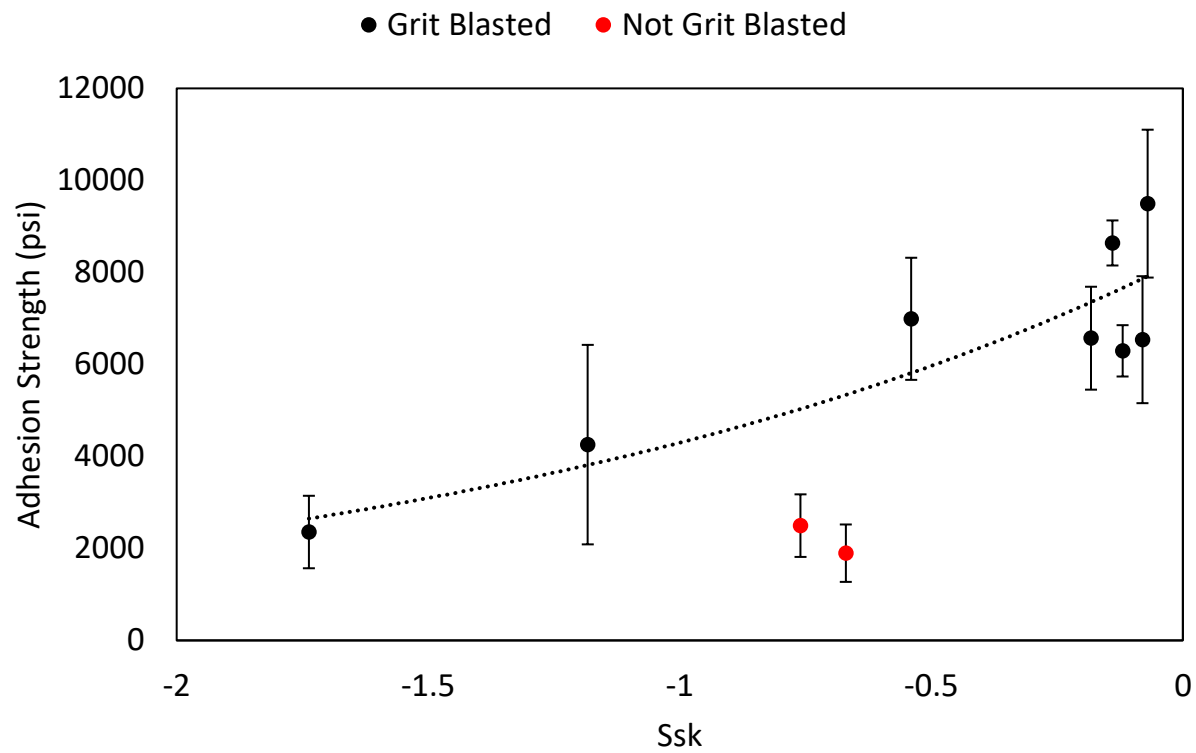


Figure 51. Si coating adhesion strength as a function of average S_{sk}

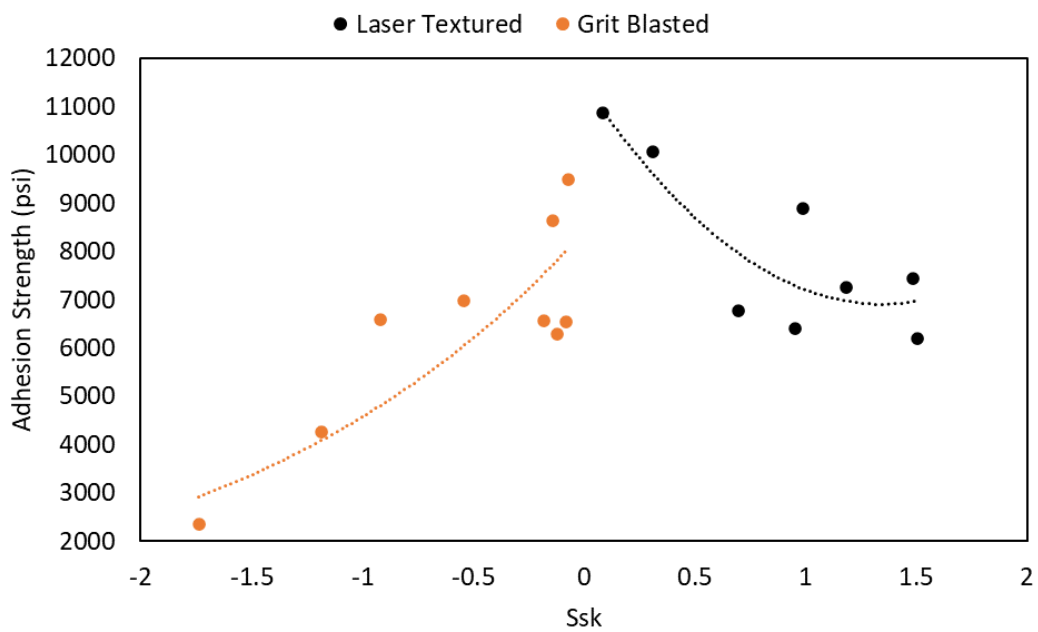


Figure 52. Si coating adhesion strength as a function of average S_{sk} for grit blasted and laser textured substrates

3.2.4 Summary

A range of surface roughness was generated on rb SiC substrates via automated grit blasting by varying blast pressure, traverse speed and step size while minimizing material/thickness loss based on previous experiments. Samples in their as-received state (not grit blasted, $S_a \sim 0.3 \mu\text{m}$) and smooth polished to $S_a < 0.1 \mu\text{m}$ were included as well. The effect of the various surface treatments on 3D surface roughness parameters was evaluated. Roughness parameters increased most substantially when blast pressure increased from 25 to 50 psi, less so from 50 to 75 psi. Notably, the skewness, S_{sk} , was negative for all samples but increased most with increasing pressure, approaching an $S_{sk} \sim 0$ where the surface has a balance of peaks and valleys. Increasing grit blasting intensity (i.e. higher pressure, slower traverse speed) results in greater average roughness but also a skewness closer to zero. Varying step size only resulted in a significant change in the parameter S_{dr} as it decreased with increasing step size.

As Si is heavily considered as a bond coating for EBCs, evaluating its bonding strength on relevant substrates and the factors that affect it is vital for optimizing these bond coatings and EBC systems. The adhesion strength of plasma-sprayed Si on these grit blasted rb SiC substrates was evaluated. Adhesion strength ranged from 1896 psi for smoother substrates to 9495 psi for rougher substrates, and quite higher values of adhesion strength were found than the only other known study [16] of plasma-sprayed Si adhesion on a Ti alloy substrate in which the measured bonding strength was reported to be 2988 ± 275 psi. Measured adhesion strength values tended to increase with increasing roughness parameter values, however one set of substrates grit blasted at a much lower traverse speed, 200 mm/s, compared to all others had higher roughness values but lower measured coating adhesion strength. The highest measured coating adhesion strength was observed for the substrates grit blasted with a small step size of 3mm, and these samples had an average skewness value closest to 0 indicating a symmetrical surface.

3.3 Laser Ablation and Si Coating Adhesion on RB SiC

One way to generate surface texture while maintaining a great deal of control over the final surface topography is via a method called laser ablation, or laser surface texturing. While this method is more time consuming, expensive and less established than other techniques like grit blasting, it provides a significant amount of control over the patterns generated on a substrate surface. The increased control this method provides has created interest in fields where surface topography is important and allows for more controlled studies of surface texture/coating relationships.

3.3.1 Process and Methods

Substrates were round, high purity, reaction bonded silicon carbide (rb SiC) buttons (M Cubed Technologies, Newark, DE, USA) with diameter of 25mm, thickness of 4.5mm, average as-received roughness (R_a) of $0.353 \pm 0.05 \mu\text{m}$ and Knoop hardness of 1080 kg/mm^2 . The buttons were cleaned with ethanol prior to laser surface texturing. To study the effect of surface texture parameters on plasma sprayed Si coating adhesion strength, a regular pattern of peaks, similar to the pattern shown in the Figure 53, with varying peak heights, widths and spacing was generated on the rb SiC substrates via pulsed laser texturing. The combination of peak heights, widths and spacings to be generated on each substrate set is provided in Table 6. For ease of discussion, a texture pattern may be referred to as height-spacing-width, so 15-30-15 corresponds to surface with peak height of $15 \mu\text{m}$, peak spacing of $30 \mu\text{m}$ and peak width of $15 \mu\text{m}$. Peak spacings were measured from the outer edge of the top surface of peaks.

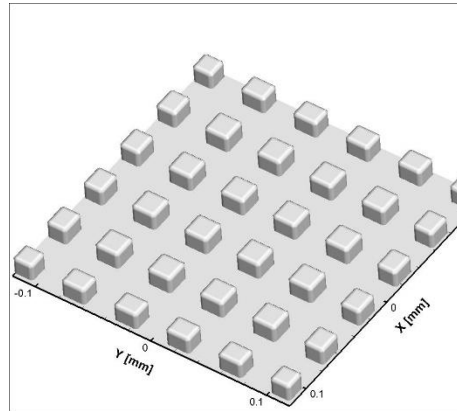


Figure 53. Simulated geometry of texture pattern generated via laser ablation on substrate surfaces

Table 6. Peak parameters for each sample laser textured pattern

Sample Pattern Set	Peak Height (μm)	Peak Spacing (μm)	Peak Width (μm)
1	15	30	15
2	15	30	30
3	30	30	15
4	30	30	30
5	15	45	15
6	15	45	30
7	30	45	15
8	30	45	30

Laser surface texturing was performed by GF Machining Solutions (Lincolnshire, IL, USA) using an AgieCharmilles Laser P 400 U machine with a 30W Ytterbium nano pulsed fiber laser. The laser parameters are shown in Table 7. The change in sample appearance after texturing and the variation in appearance with differing peak pattern parameters can be seen in Figure 54.

Table 7. Laser parameters used for generating surface texture

Laser Parameter	Value
Power	14 %
Frequency	40 kHz
Line Speed	600 mm/s
Pulse Duration	50 ns
Focal Lens	100 mm

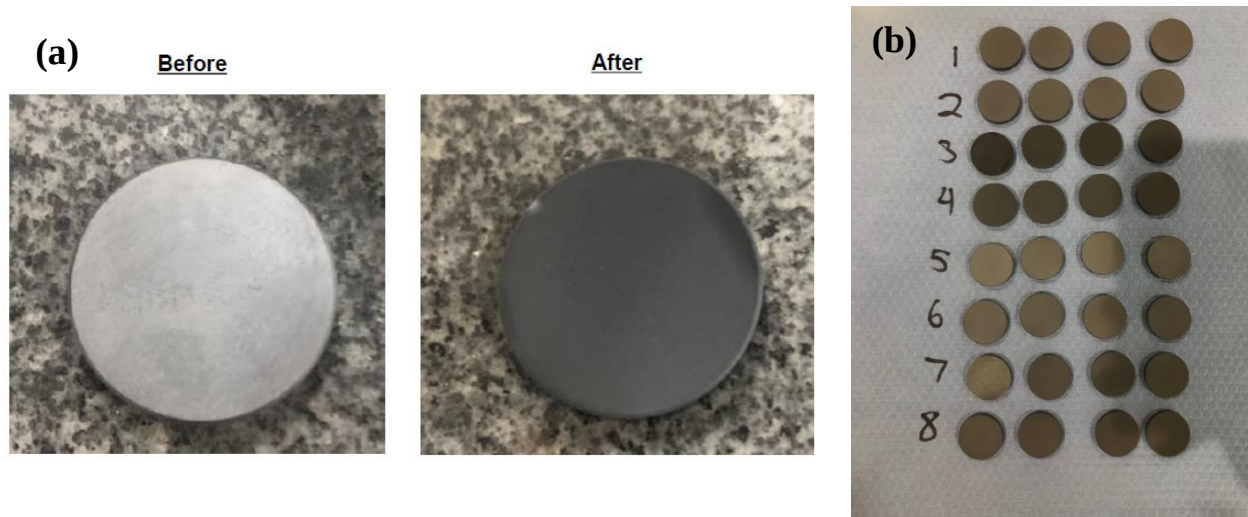


Figure 54. Photographs of rb SiC buttons a) before and after laser texturing and b) variation in surface appearance with texture pattern

To characterize the textured surfaces, an Olympus LEXT OLS4100 confocal laser scanning microscope was used to obtain 2D and 3D images of the substrate surface, measure 3D surface roughness and 2D surface profiles. Laser-scanning microscope images and 3D surface maps of the generated patterns for each sample set are shown in Figure 55 and 57, respectively, and clearly illustrate the differences between the texture patterns. Texture parameters, peak height, width and spacing, were measured on one sample from each sample set. Three to four measurements of each parameter were taken in three different locations on the sample surface and means and standard deviations were calculated from ten measurements total, as shown in Table 8. Mean peak dimensions are also plotted for comparison in Figure 56. Measured 3D surface roughness values, along with peak height to spacing ratios, for each sample pattern set are given in Table 9. All roughness parameters were measured in three different locations on the sample surface using a 50x objective lens with a scan area of 260x260 μm and cutoff value, λ_c , of 800 μm and then averaged.

Detailed descriptions of 3D roughness parameters are provided in ISO 25178 specifications and by Deltombe et al. [89-90]. Parameters of interest are the amplitude parameters S_a , the arithmetic mean height, and S_{sk} , skewness, the functional parameter S_k , the core roughness depth, and the hybrid parameter S_{dr} , the developed interfacial area ratio. S_a provides a general measure of the average roughness of a surface similar to the commonly used parameter R_a , while S_{sk} describes the symmetry of a surface and gives an idea of whether the

surface is dominated by peaks or valleys. S_k is a measure of the “core roughness” of a surface with the tallest peaks and deepest valleys removed. S_{dr} provides a measure of the amount of additional surface area contributed by the surface texture compared to a smooth surface.

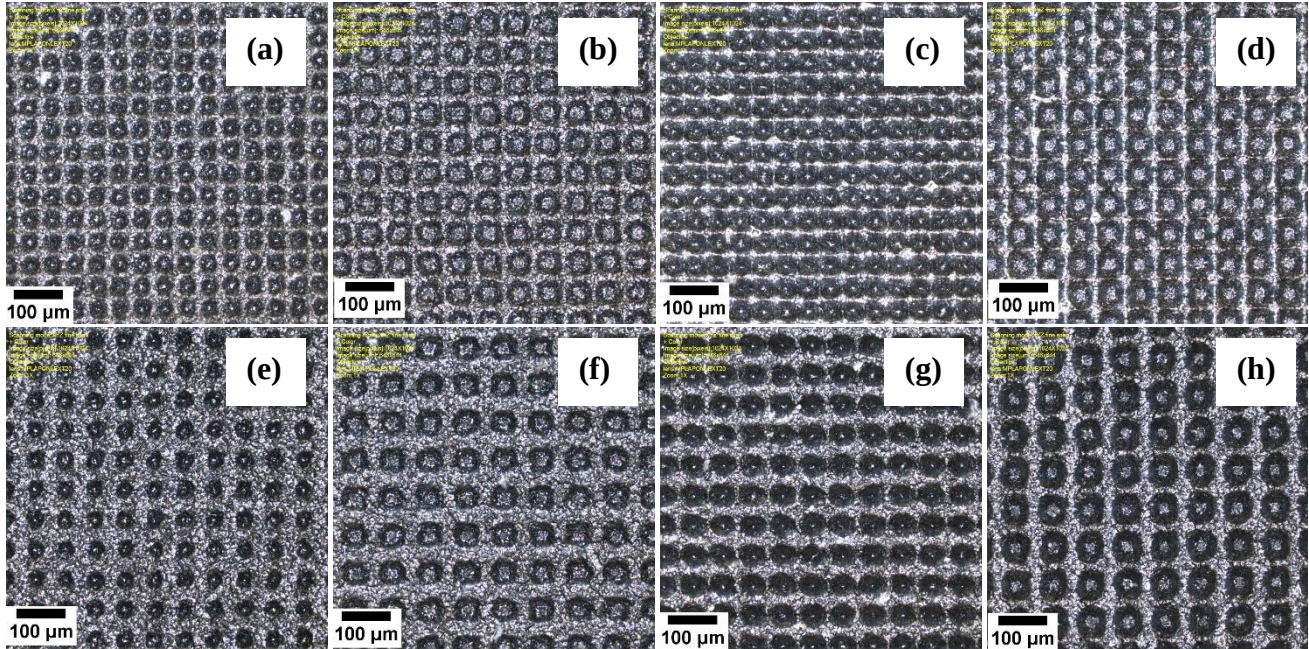


Figure 55. Laser scanning microscope images of surface texture for each sample pattern: a) 15-30-15, b) 15-30-30, c) 30-30-15, d) 30-30-30, e) 15-45-15, f) 15-45-30, g) 30-45-15, h) 30-45-30

Table 8. Measured means and standard deviations of peak dimensions for each texture pattern

Sample Set		Peak Height	Peak Spacing	Peak Width
1	(Mean)	18.17	30.61	14.5
(15-30-15)	(SD)	1.38	1.34	1.03
2		15.88	29.83	30.5
(15-30-30)		0.95	1.24	1.16
3		29.83	28.84	15.77
(30-30-15)		3.08	1.7	0.92
4		29.33	31.32	29.03
(30-30-30)		0.9	1.25	0.93
5		16.25	44.73	14.86
(15-45-15)		1.06	1.4	0.5
6		17.09	44.32	30.28
(15-45-30)		0.94	1.59	1.5
7		33.91	44.63	15.03
(30-45-15)		2.45	1.1	1.07
8		30.59	43.42	31.18
(30-45-30)		1.18	2.08	1.86

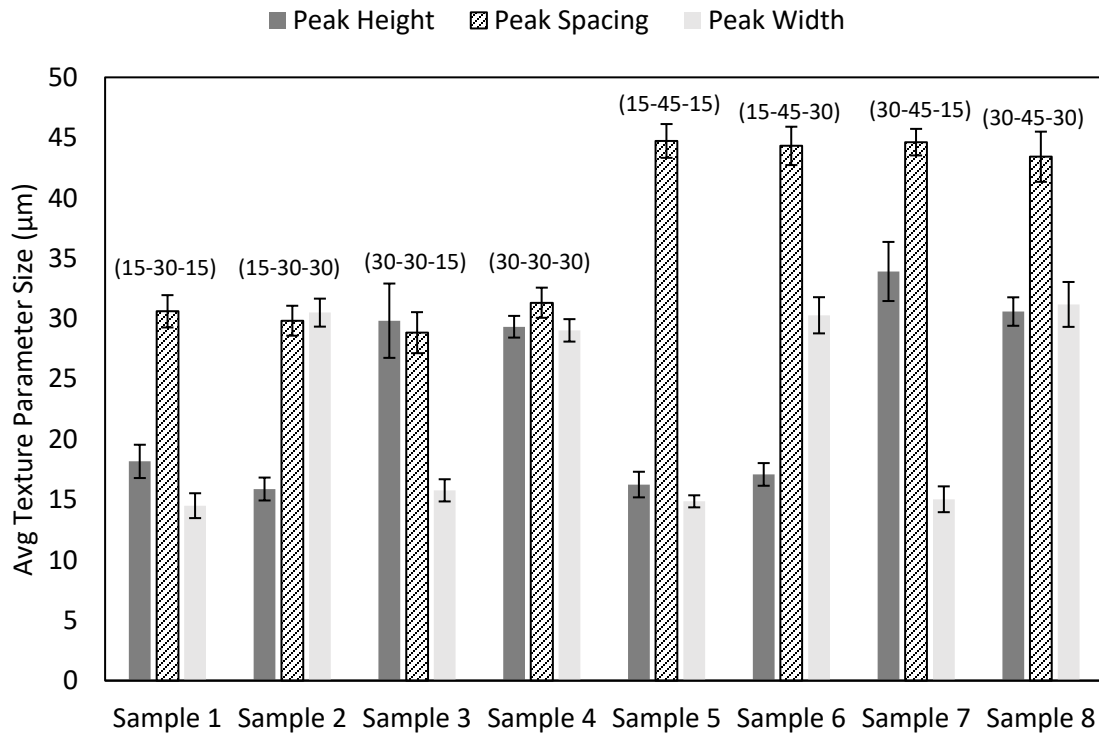


Figure 56. Average peak dimensions for each sample pattern along with standard deviations represented by error bars

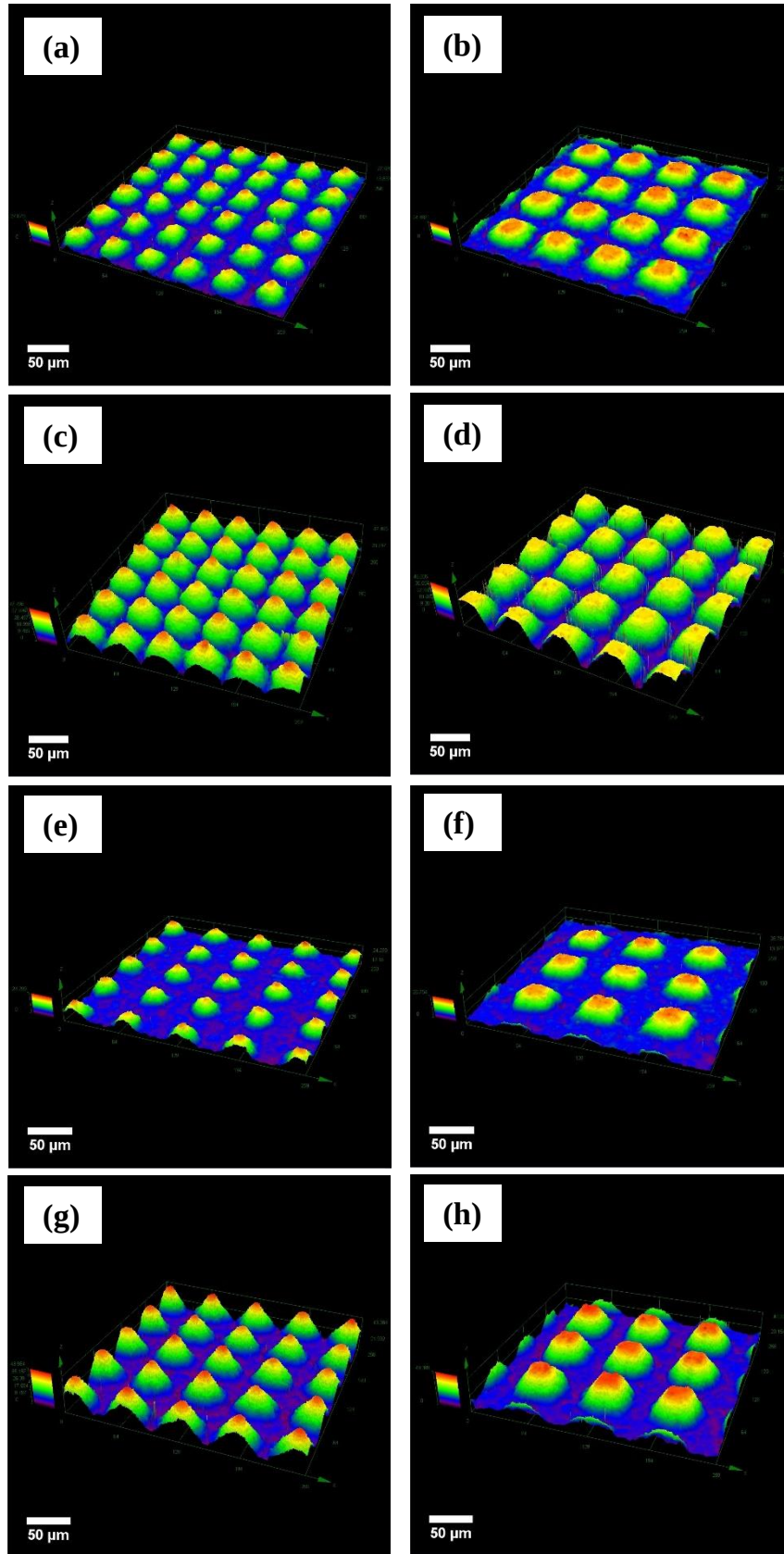


Figure 57. Laser scanning microscope generated 3D topographical maps of surface texture for each sample pattern: a) 15-30-15, b) 15-30-30, c) 30-30-15, d) 30-30-30, e) 15-45-15, f) 15-45-30, g) 30-45-15, h) 30-45-30

Table 9. Average 3D surface roughness parameters and standard deviations for each texture pattern

Sample Set		Sq[μm]	Ssk	Sku	Sp[μm]	Sv[μm]	Sz[μm]	Sa[μm]
1	(Mean)	5.16	0.95	2.99	20.50	9.83	30.33	4.25
	(SD)	0.22	0.10	0.34	3.20	0.59	2.64	0.26
2		5.75	0.70	2.09	13.96	10.48	24.44	4.99
		0.06	0.04	0.06	0.77	0.41	1.19	0.07
3		9.75	0.31	2.19	26.82	21.63	48.46	8.17
		0.06	0.02	0.03	0.64	0.66	1.30	0.07
4		10.00	0.09	1.70	19.13	20.94	40.07	8.74
		0.12	0.04	0.03	0.27	0.73	0.84	0.14
5		4.23	1.49	4.60	18.49	8.67	27.16	3.19
		0.16	0.16	0.34	1.32	1.56	2.76	0.15
6		5.12	1.51	4.07	17.37	8.64	26.01	3.98
		0.06	0.03	0.10	0.29	0.61	0.64	0.07
7		9.70	0.99	2.85	29.09	15.65	44.75	8.11
		0.10	0.01	0.01	1.17	0.54	1.68	0.09
8		9.33	1.18	3.18	27.02	13.55	40.57	7.66
		0.09	0.02	0.06	1.16	0.83	0.34	0.11
Manually GB		1.26	-1.15	4.69	4.30	7.85	12.14	0.99
		0.34	0.15	0.78	0.20	1.07	0.96	0.28

Sample Set		Sk[μm]	Spk[μm]	Svk[μm]	Sal[μm]	Str	Sdr	Height/Spacing Ratio
1	(Mean)	7.60	9.93	1.14	12.16	0.78	189.00	0.5
	(SD)	0.11	0.42	0.06	0.25	0.03	18.63	
2		6.91	12.23	1.39	18.98	0.93	96.66	0.5
		0.53	0.41	0.20	0.13	0.01	5.57	
3		28.48	7.67	2.21	12.47	0.34	232.30	1
		1.63	0.57	0.33	0.09	0.32	7.55	
4		30.07	2.86	1.88	17.76	0.67	369.60	1
		1.67	1.65	0.21	0.13	0.03	46.70	
5		5.04	10.49	1.34	16.93	0.90	82.20	0.33
		0.91	0.11	0.28	0.72	0.02	4.52	
6		3.72	14.07	1.08	22.50	0.96	82.80	0.33
		0.06	0.13	0.16	0.39	0.00	2.26	
7		8.74	21.58	1.81	17.94	0.83	236.16	0.66
		0.20	0.35	0.19	0.19	0.02	32.58	
8		6.89	22.77	1.85	22.12	0.85	146.86	0.66
		0.38	0.55	0.15	0.32	0.01	4.02	
Manually GB		2.56	0.38	2.09	16.34	0.84	23.20	
		0.90	0.04	0.41	4.48	0.08	1.50	

Prior to depositing the Si coating via APS, samples were preheated via two traverses of the plasma torch without powder with a standoff distance of 130 mm and traverse rate of 500 mm/s. Based on previous measurements utilizing the same preheating procedure, samples were expected to be between 140 and 150 °C when coating began. The spraying powder used was pure silicon, HA 9197-2 from HAI Advanced Material Specialists, with a mean particle size of 71 μm . Three samples per texture pattern were preheated and coated simultaneously to keep spray parameters consistent for all samples. Substrates were coated via 11 total passes of the plasma torch to reach a coating thickness of 200 μm . Spray parameters are shown in Table 9 and the spraying setup inside the plasma spray booth at the Commonwealth Center for Advanced Manufacturing (CCAM) is shown in Figure 58.

Table 10. Plasma spray parameters use for depositing silicon coating

Spraying Parameter	Value
Spray Gun	F4MB-XL (Oerlikon Metco)
Current, A	506
Voltage, V	73.5
Argon gas flow, NLPM	57
Hydrogen gas flow, NLPM	9
Powder gas flow, NLPM	7
Powder feed rate, g/min	29
Traverse rate, mm/s	500
Standoff distance, mm	130
Particle temperature, °C	2508
Particle velocity, m/s	330



Figure 58. Plasma spraying setup used at CCAM

The adhesion strength of Si coating on rb SiC substrates was measured via tensile test according to ASTM C633 [91]. The cylindrical rb SiC buttons were joined to grit blasted tensile test bars using FM1000 epoxy, which were cured at 192 °C for 3.25 hours. The bottom, non-coated side of samples were grit blasted prior to attachment with 220 SiC grit to help ensure sufficient epoxy adhesion. Tensile adhesion tests were carried out with a head rate of 1 mm/min and three coated samples per sample set were tested and results averaged.

3.3.2 Results and Discussion

The average coating adhesion strength for each sample set, including samples manually grit blasted (MB) directly prior to spraying for comparison, is plotted in Figure 59, with error bars representing plus or minus one standard deviation. The highest average coating adhesion strength was observed for the 30-30-15 and 30-30-30 texture patterns and average adhesion strength on the manually grit blasted substrates was close to that of the laser textured substrates with a high variation between samples. Interestingly, the smallest variation in adhesion strength values was seen for the sample set with the highest average, 30-30-30.

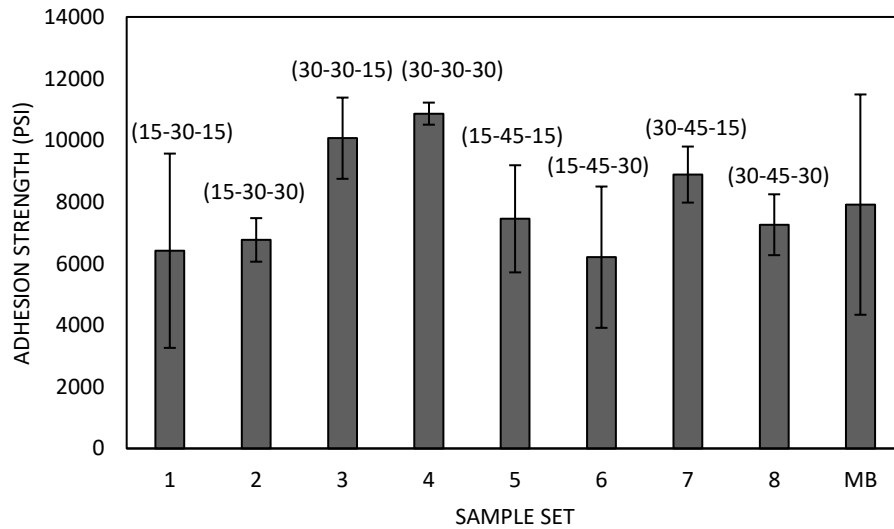


Figure 59. Average plasma sprayed silicon coating adhesion strength for each surface texture pattern. Labels correspond to (peak height-peak spacing-peak width)

The plots in Figure 60 show the effect of each individual texture parameter on coating adhesion strength when the other two parameters are held constant. In Figure 60a, average adhesion strength is seen to increase with peak height in each case, with the most significant differences seen for samples where the peak spacing was 30 μm . Differences in adhesion strength with changing peak width, shown in Figure 60b, were much less substantial. The average adhesion strength increased with peak width when the spacing was 30 μm , decreased with peak width when the spacing was 45 μm and the most significant change was observed when peak height was 30 μm and spacing was 45 μm and peak width was increased. A possible explanation for this is the increase in plain area on the surface with increased width, along with

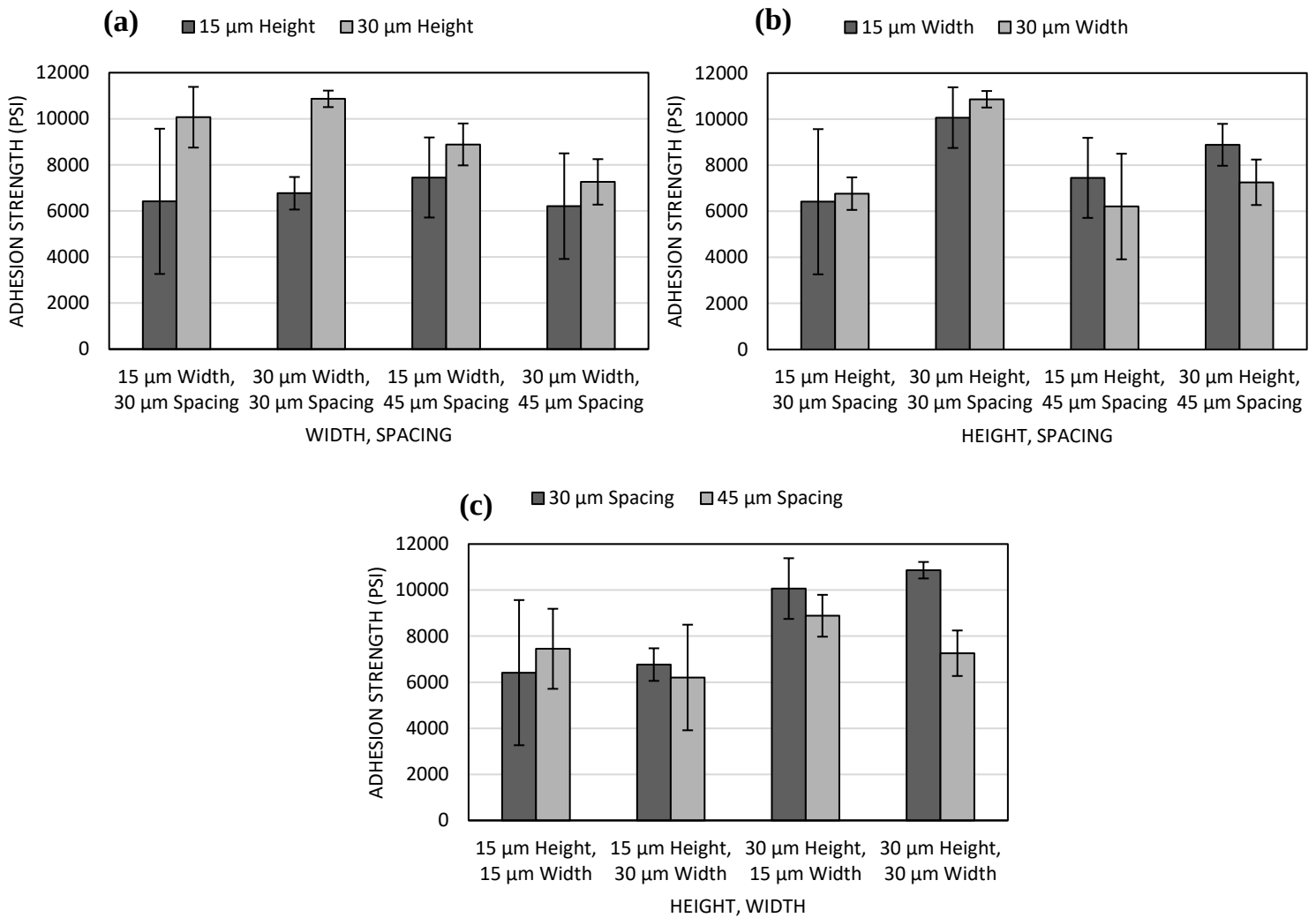


Figure 60. Plots showing the effect of a) peak height, b) peak width and c) peak spacing on the average coating adhesion strength for each surface texture

greater plain area present with higher spacing reducing the degree of interlocking between coating and substrate. The effect of peak spacing is shown in Figure 60c and the differences in adhesion strength with varying spacing are not substantial considering the variation, except for when peak height and width were both 30 μm . This, once again, could be related to the increased plain area realized when spacing is increased, reducing mechanical interlocking, along with increased interlocking likely with tall, closely-spaced peaks of the 30-30-30 texture, hinting at the possible role of the peak height to spacing ratio.

The ratio of texture height to spacing is plotted with average coating adhesion strength for each texture pattern in Figure 61. The patterns with the highest height/spacing ratio of 1, patterns 3 (30-30-15) and 4 (30-30-30), also have the highest average adhesion strength. The pattern with the third highest average adhesion strength, pattern 7 (30-45-15), had a height/spacing ratio of 0.66, one step below a ratio of 1 for this set of samples. The average adhesion strength as a function of height/spacing ratio is plotted in Figure 62. While no difference in average adhesion strength is observed between a 0.33 and 0.5 height/spacing ratio, it is seen to increase for height/spacing ratios of 0.66 and 1. Average adhesion strength increased substantially when height/spacing ratio increased to 1.

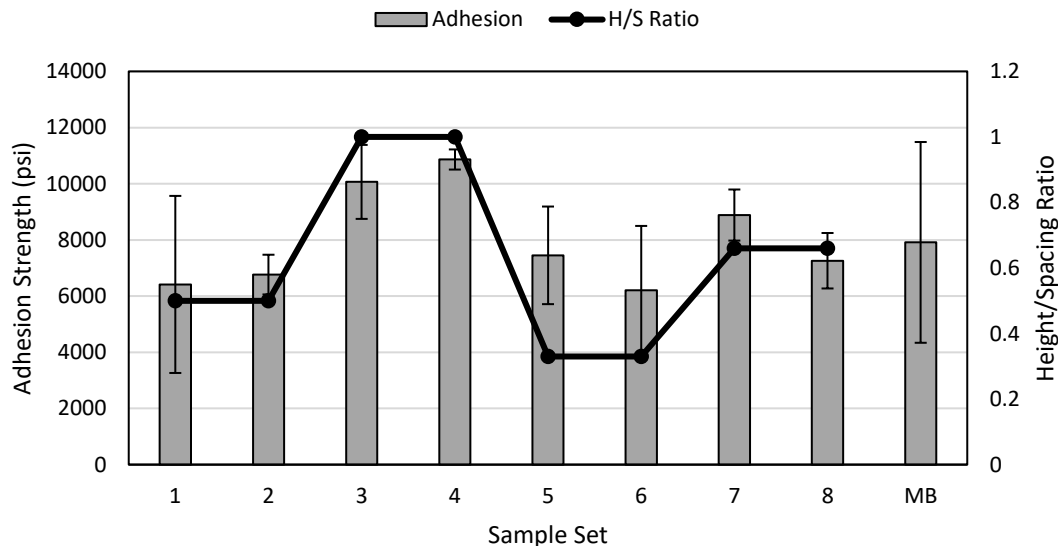


Figure 61. The average coating adhesion strength plotted with the corresponding peak height to spacing ratio for each texture pattern set

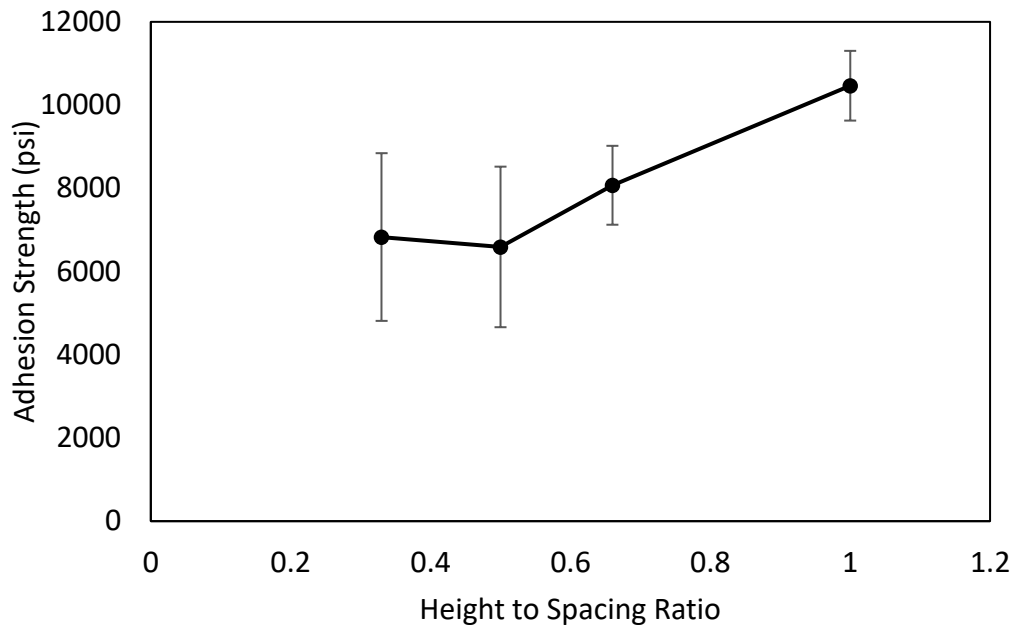


Figure 62. The average coating adhesion strength as a function of peak height to spacing ratio

Figure 63 shows average adhesion strength and average S_a plotted together for each texture pattern allowing for comparison between sets and observation of trends within samples with the same height/spacing ratio or texture parameters. The three sample sets with the highest average adhesion strength corresponded with the texture patterns with the highest average S_a . With the exception of samples with the smallest height/spacing ratio of 0.33, sets 5 and 6, average adhesion strength increased with S_a for samples with the same height/spacing ratio. The average S_a of the manually grit blasted samples was quite lower than that of the laser textured samples, but the adhesion strength was similar between grit blasted and laser textured surfaces. This is likely due to the very different nature of the surfaces and the differences in the the plain area on the surface of grit blasted and pattern textured substrates as suggested by others [54]. Adhesion strength is plotted as a function of S_a in Figure 64. For the laser textured samples, S_a and adhesion strength look to be unrelated at lower values of S_a , which also correspond to lower height/spacing ratios. At higher values of S_a and height/spacing ratios, adhesion strength increases with S_a . Once again, the likely increase in plain area and decrease in texture area for

mechanical interlocking at lower height/spacing ratios could contribute detrimentally to coating adhesion despite the average roughness, S_a .

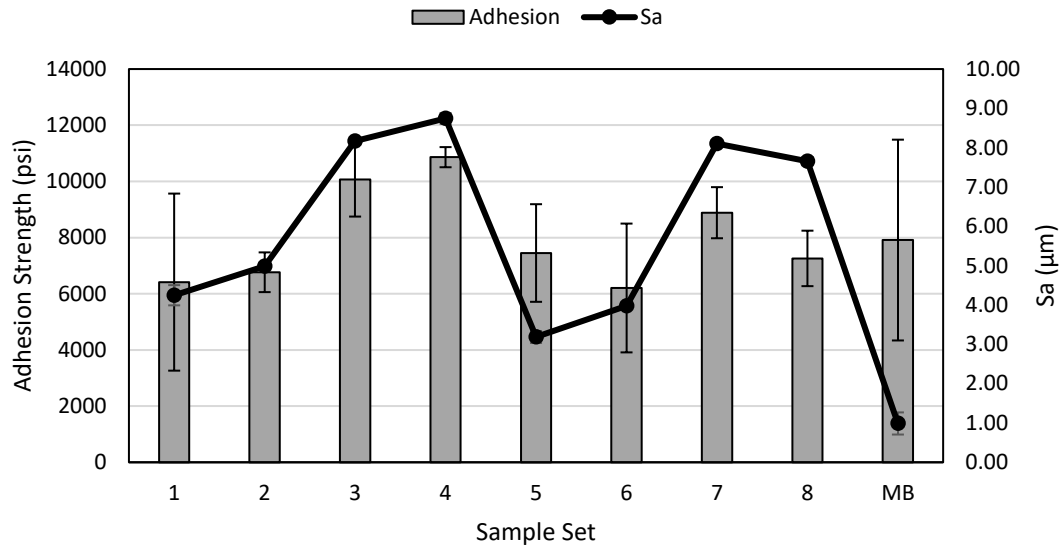


Figure 63. The average coating adhesion strength plotted with the corresponding average 3D roughness S_a for each texture pattern set

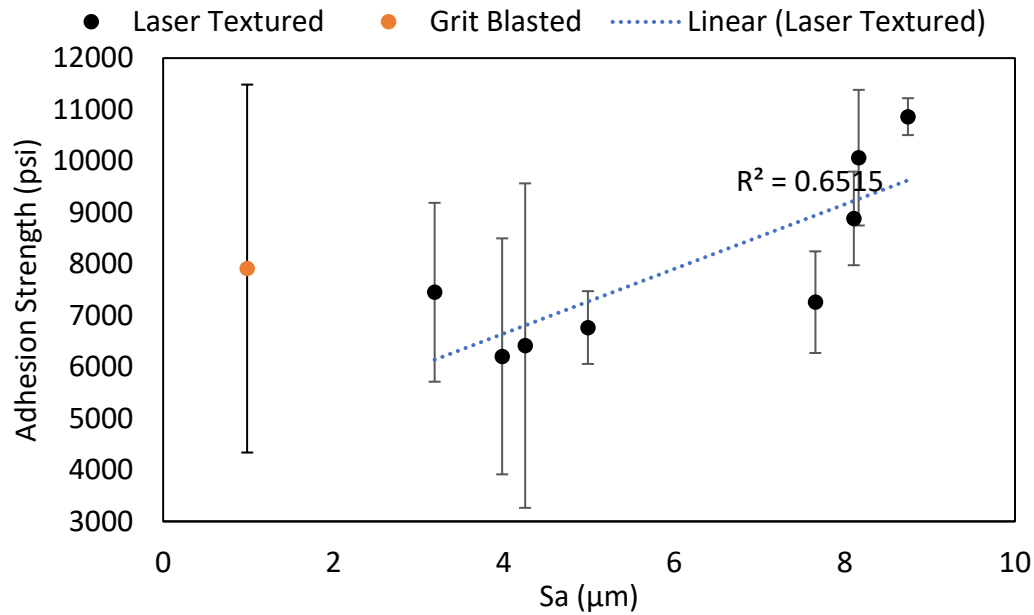


Figure 64. The average coating adhesion strength as a function of S_a

The average adhesion strength of a plasma-sprayed Si coating on rb SiC substrates as a function of S_a is plotted for grit blasted and laser textured substrates in Figure 65.

S_a for the grit blasted substrates is considerably lower than the textured samples and adhesion strength increases drastically when S_a increases above $1\text{ }\mu\text{m}$ from around $0.6\text{ }\mu\text{m}$ to $1.3\text{ }\mu\text{m}$. Adhesion strength then seems to plateau as S_a increases, even for laser textured samples, until it reaches close to $8\text{ }\mu\text{m}$ when adhesion strength increases with S_a again to the maximum observed value.

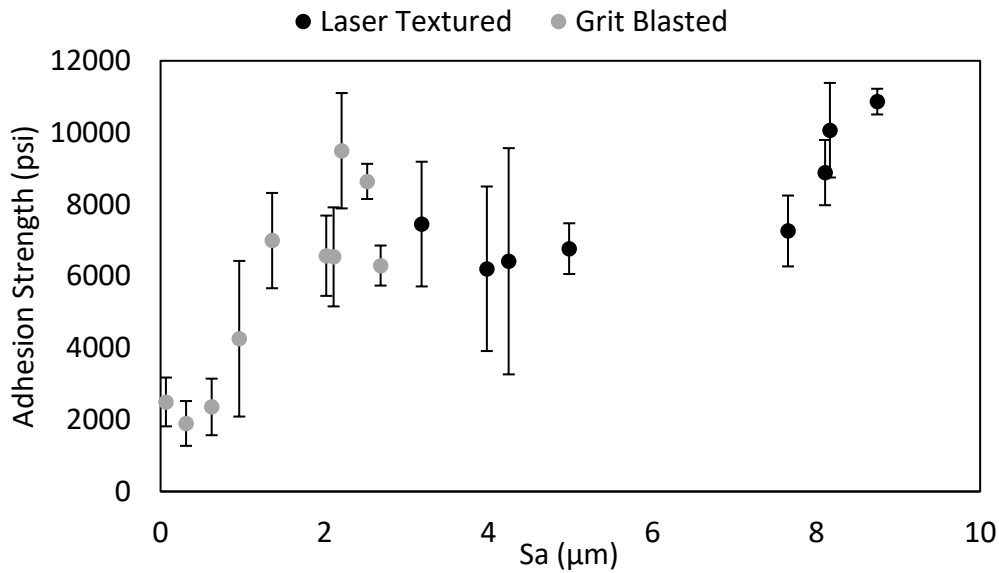


Figure 65. The average coating adhesion strength as a function of S_a for grit blasted and laser textured substrates

Average coating adhesion strength is plotted with average S_k for each pattern in Figure 66 and as a function of S_k in Figure 67. Very high values of S_k are observed for sample patterns 3 (30-30-15) and 4 (30-30-30), which also correspond to the highest height/spacing ratio and average adhesion strength values. It is clear that the dramatic increase in S_k corresponds with a dramatic increase in adhesion strength as well for these samples. For samples with lower measured adhesion strength values, S_k is similar just as the average adhesion strengths are between this group, but changes in adhesion strength tend to correspond to changes in this roughness parameter. High values of S_k likely indicate a surface which promotes a high degree of interlocking between coating and substrate.

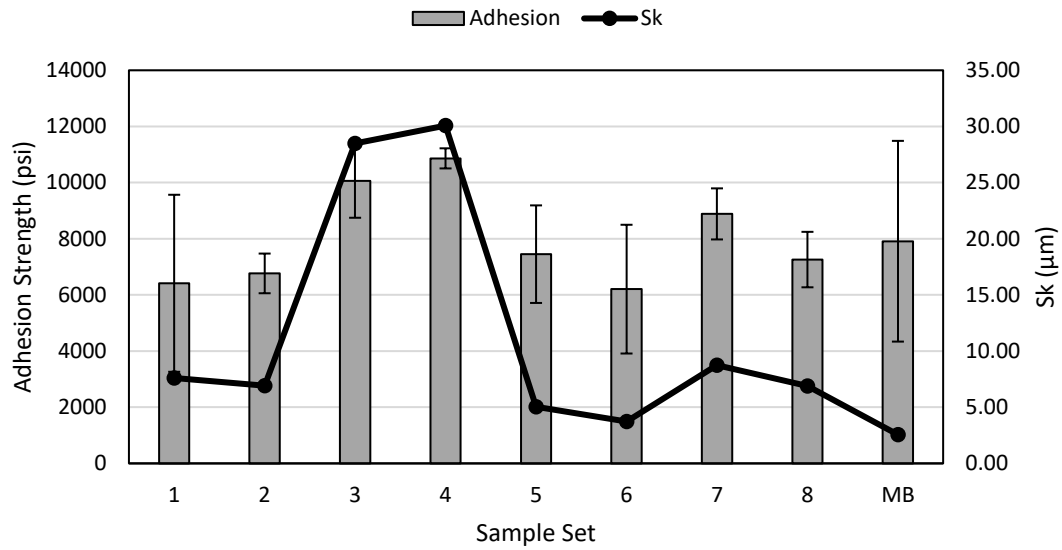


Figure 66. The average coating adhesion strength plotted with the corresponding average S_k for each texture pattern set

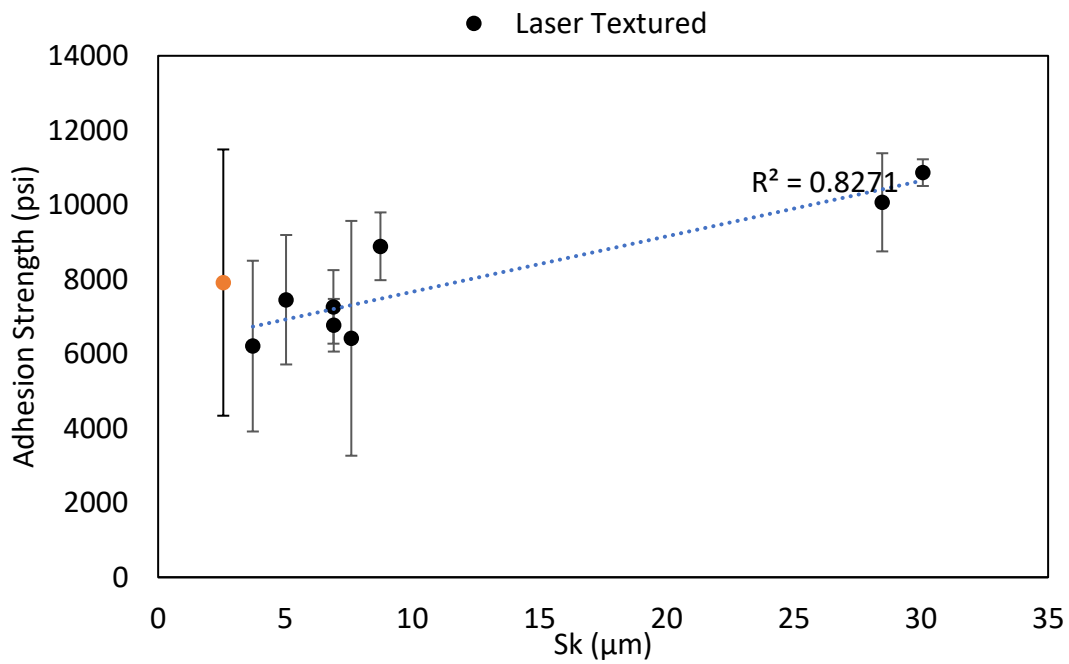


Figure 67. The average coating adhesion strength as a function of S_k

Similar to other parameters, the highest measured value of Sdr corresponds to the highest measured adhesion strength for the laser textured substrates as seen in Figures 68 and 69. Adhesion strength shows an increasing tendency with Sdr, however changes in Sdr don't always correspond to changes in average adhesion strength. As Sdr represents the additional surface area contributed by texture, it is expected to relate to the degree of contact between a substrate and

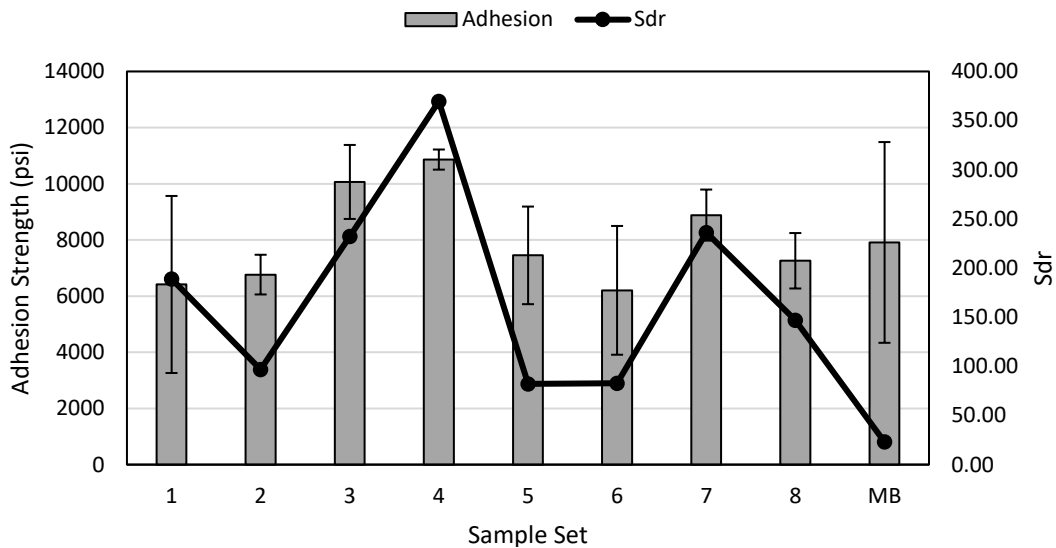


Figure 68. The average coating adhesion strength plotted with the corresponding average Sdr for each texture pattern set

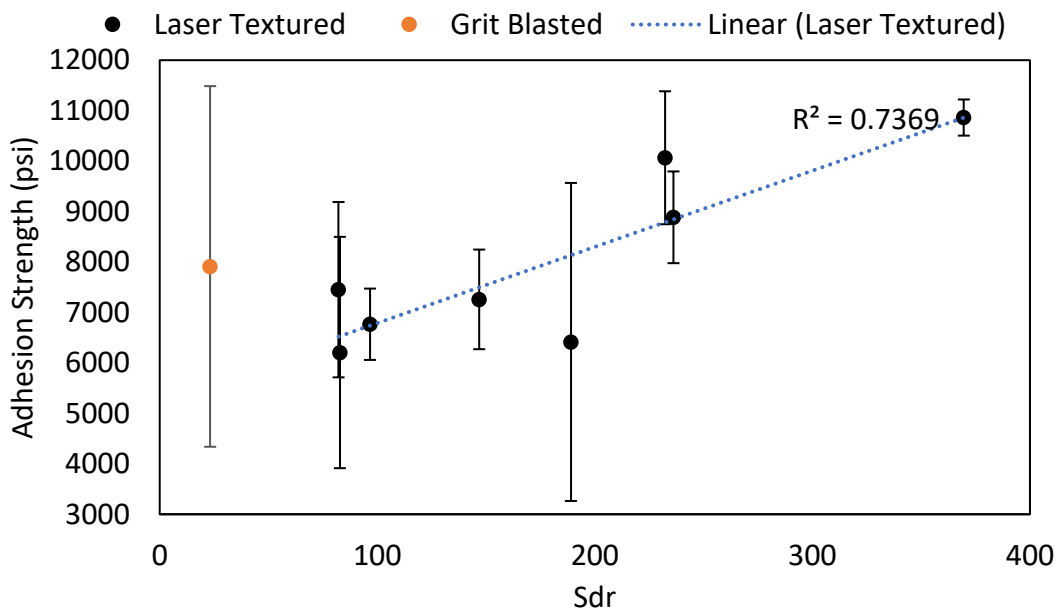


Figure 69. The average coating adhesion strength as a function of Sdr

sprayed coating, however these results seem to suggest other factors, like cooling rates, could contribute to coating adhesion strength.

Adhesion strength is plotted as a function of the skewness parameter S_{sk} for laser textured and previously grit blasted and coated rb SiC substrates in the Figure 70. S_{sk} tends to be positive, indicating a greater amount of surface peaks than valleys, for the laser textured surfaces and negative, indicating more valleys than peaks, for grit blasted surfaces. Coating adhesion strength is seen to increase as the skewness approaches zero for both types of surface treatment, indicating that a surface which is more balanced in terms of the amount of peaks and valleys is ideal for improving the adhesion of a plasma-sprayed coating. A good balance of surface peaks and valleys on which a coating is plasma sprayed will likely promote mixed-mode failure of the coating and improve the coating adherence, as suggested by Kromer et al. [52, 56, 92-93].

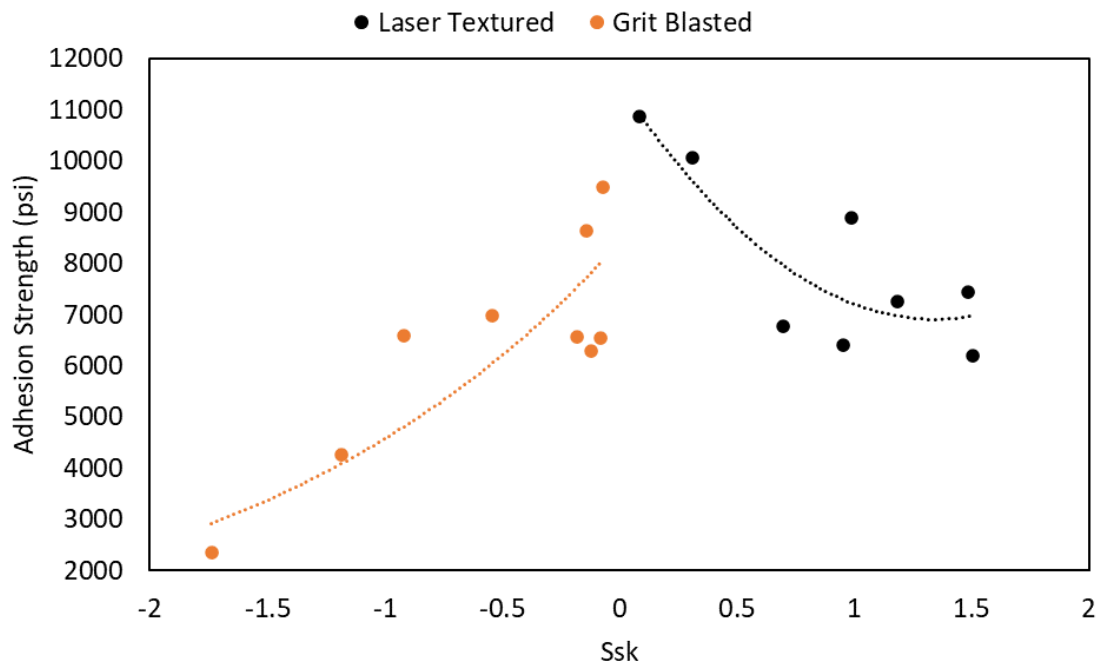


Figure 70. The average coating adhesion strength as a function of S_{sk} for laser textured and grit blasted rb SiC coated with plasma sprayed silicon

3.3.3 Summary

A laser ablation technique was utilized to generate regularly patterned texture on rb SiC substrates. The patterns consisted of peaks/posts on the surface with varying height, spacing between peaks and width. Each of these peak parameters were varied at two levels in order to investigate their effect on the adhesion strength of a plasma-sprayed Si coating. Average adhesion strength values for these laser textured substrates were all higher than rb SiC substrates with an $R_a < 1 \mu\text{m}$ from the previous study of Si coating adhesion strength on grit blasted samples and had a minimum value of 6206 psi. Adhesion strength on laser textured surfaces tended to be similar to that of grit blasted substrates blasted at 50 psi and higher, though a notable increase in adhesion strength from the grit blasted substrates was observed for the two laser ablated sets with the highest average adhesion strength values. Height/spacing ratio of the texture patterns was observed to be an indicator of coating adhesion strength, as well as S_a , S_k , S_{dr} and S_{sk} . The amount of plain area on the laser textured surfaces likely was a factor in coating adhesion as well. Unlike the grit blasted substrates, the laser textured surfaces all had positive skewness values, S_{sk} , however, like the grit blasted substrates, coating adhesion strength increased as skewness approached 0. A balanced, symmetrical but still rough surface contributes to increased adhesion strength of plasma sprayed Si on a rb SiC substrate.

3.4 Si Particle Size and Substrate Preheating Effect on Si Adhesion and Splats

3.4.1 Process and Methods

3.4.1.1 Si Particle Size

The effect of Si powder mean particle size on plasma-sprayed coating adhesion strength was studied by varying the particle size distribution of sprayed Si coatings on grit blasted rb SiC substrates. As-received Si powder with a mean particle size of $71.3 \mu\text{m}$ was sieved to generate the three different powder particle size distributions shown in Figure 71. The sets of sieved Si powder had mean particle diameters of 56, 73 and $94 \mu\text{m}$.

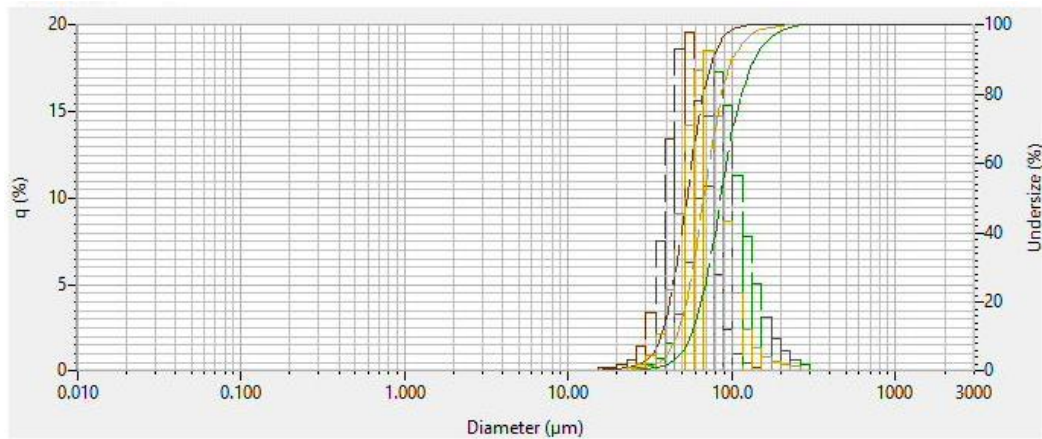


Figure 71. Particle size distributions of three sets of Si powder sieved to mean particle diameters of 56, 73 and 94 μm

Substrates were all grit blasted via the same method and blasting parameters in order to generate comparable surface roughness. Substrates sprayed with Si with mean particle size of 56, 73 and 94 μm had average S_a values of $1.98 \pm 0.072 \mu\text{m}$, $2.16 \pm 0.16 \mu\text{m}$ and $2.11 \pm 0.18 \mu\text{m}$, average S_{dr} values of 37.2 ± 1.4 , 36.06 ± 0.52 , 36.6 ± 1.07 and average S_{sk} values of -0.085 ± 0.075 , -0.11 ± 0.35 and -0.08 ± 0.15 , respectively. Prior to depositing the Si coating via APS, samples were preheated via two traverses of the plasma torch without powder. A set of five samples at a time were preheated and coated simultaneously for each particle size distribution, and all plasma spray operating conditions besides particle size were kept constant to observe its effect on coating adhesion strength. Table 11 provides the operating conditions and measured in-flight particle parameters for plasma spraying of Si for each mean particle size.

Table 11. Spray parameters for plasma spraying Si with varied mean particle size

Spraying Parameter	56 μm	73 μm	94 μm
Spray Gun	F4MB-XL (Oerlikon Metco)		
Current, A	506	507	506
Voltage, V	74.3	73.8	74
Argon gas flow, NLPM	57	57	57
Hydrogen gas flow, NLPM	9	9	9
Powder gas flow, NLPM	7	7	7

Powder feed rate, g/min	29	29	29
Traverse rate, mm/s	500	500	500
Standoff distance, mm	130	130	130
Particle temperature, °C	2321	2359	2466
Particle velocity, m/s	368	342	329

3.4.1.2 Substrate Preheating

Three levels of preheating and corresponding substrate temperature were used to observe the effect of preheated substrate temperature on Si coating adhesion strength. The number of preheat passes prior to coating and corresponding measured substrate temperature is shown in Table 12.

Table 12. Number of passes of plasma torch and corresponding substrate temperature for each preheat level

# of Preheat Passes	Substrate Temperature
0	32° C
2	115° C
30	350° C

One set of samples underwent no preheating and were coated directly at ambient temperature around 32°C. The second and third set of samples underwent two and thirty preheat passes of the plasma torch, reaching approximately 115°C and 350°C respectively just before coating began. Prior to depositing the Si coating via APS, the plasma torch (Figure 72b) without powder was traversed across the substrates with a varying number of total passes to generate different substrate temperatures and levels of preheating. Five samples at a time were preheated and coated simultaneously at each preheat level. Substrate temperature was measured during the preheating and coating process via a K type thermocouple placed on the back of a single samples as seen in Figure 72a. The measured substrate temperature during preheating and coating for each sample set is plotted in Figures 73-75. The temperature of the other four samples were assumed to be the same as each step of the process and time in between was consistent. In addition, one sample for each temperature was preheated individually and not coated for subsequent x-ray photoelectron spectroscopy (XPS) analysis using a PHI Quantera SXM for quantitative analysis of the top few nanometers of the sample surface. Following preheating, these samples were stored together until XPS could be performed.



Figure 72. Photo of a) thermocouple placement within sample holder and b) the plasma spray gun used for preheating and coating samples

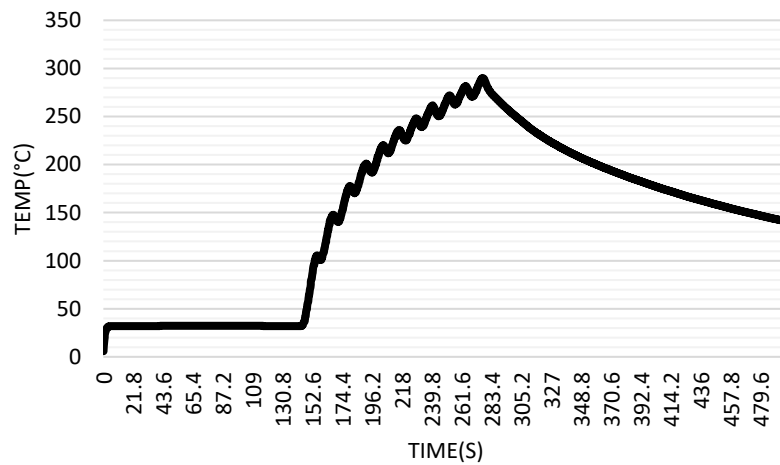


Figure 73. Plot of measured substrate temperature over time during coating of a room temperature substrate

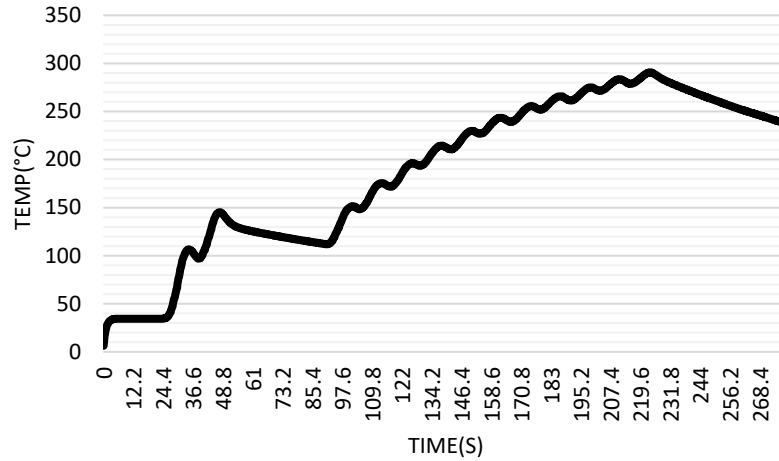


Figure 74. Plot of measured substrate temperature over time during preheating via two passes of the plasma torch and subsequent coating

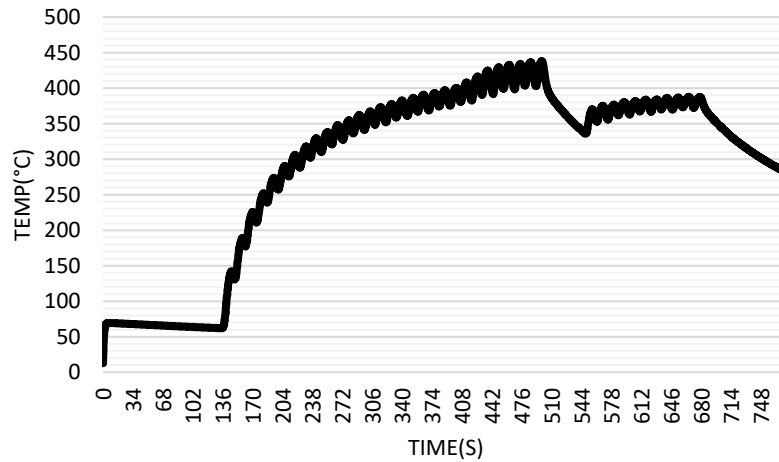


Figure 75. Plot of measured substrate temperature over time during preheating via thirty passes of the plasma torch and subsequent coating

Substrates were all grit blasted via the same method and blasting parameters in order to generate comparable surface roughness and roughness parameters were measured prior to preheating and coating. Mean values of R_a , R_{sm} and $R_{\Delta q}$ for substrates at each corresponding preheat temperature are plotted in Figures 76-78. Table 13 displays means and standard deviations of measured roughness parameters for substrates at each preheat level.

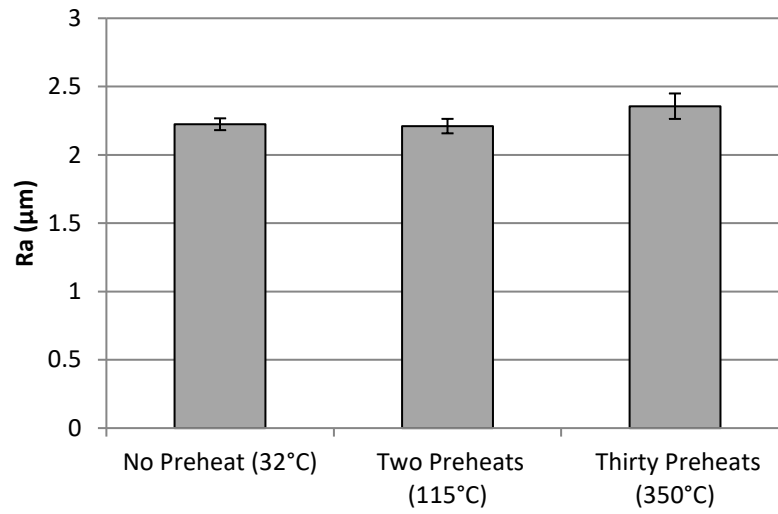


Figure 76. Average Ra of substrates coated at different preheat temperature

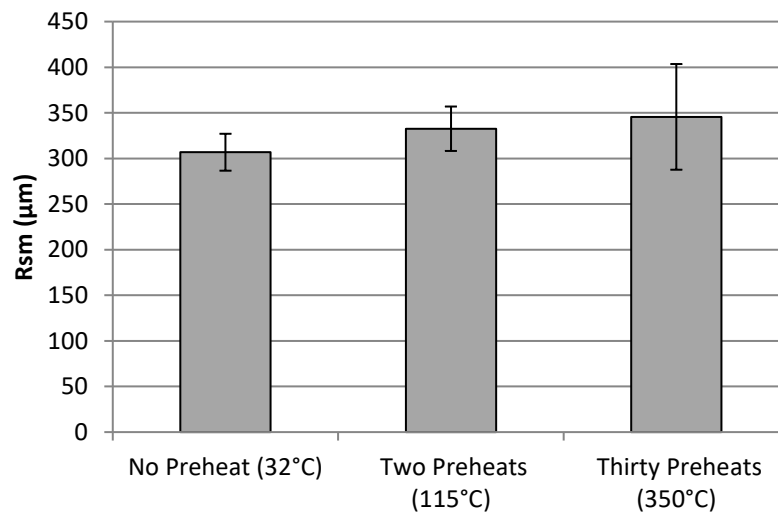


Figure 77. Average Rsm of substrates coated at different preheat temperature

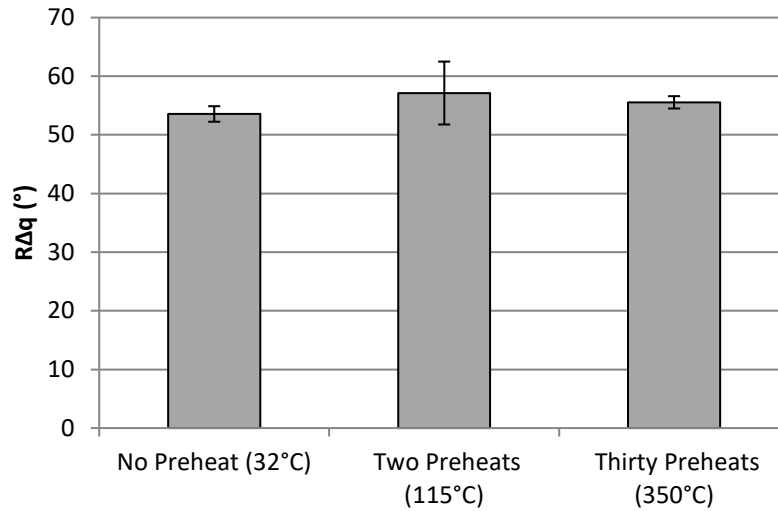


Figure 78. Average $R\Delta q$ of substrates coated at different preheat temperature

Table 13. 2D and 3D roughness parameters of substrates coated at different preheat temperature

Sub.								
# of preheats	Temp		Rp[μm]	Rv[μm]	Rz[μm]	Rc[μm]	Rt[μm]	Ra[μm]
No Preheat	32°C	Mean	13.130	11.036	24.166	12.645	40.013	2.224
		SD	6.017	0.718	5.319	0.124	16.146	0.062
Two Preheats	115°C	Mean	21.593	10.238	31.830	13.002	86.952	2.211
		SD	14.527	0.303	14.461	0.242	72.931	0.043
Thirty Preheats	350°C	Mean	8.899	16.989	25.888	14.123	52.804	2.356
		SD	0.403	6.001	6.052	1.582	26.866	0.093

# of preheats	Sub. Temp		Rq[μm]	Rsk	Rku	Rsm[μm]	RΔq[°]	Rδc[μm]	Rmr[%]	Rzjis[μm]
No Preheat	32°C			-						
		Mean	2.83	0.27	6.45	306.79	53.56	4.59	100.00	16.65
		SD	0.06	0.30	5.05	20.22	1.32	0.21	0.00	0.67

Two Preheats	115°C	Mean	2.85	0.88	54.96	332.54	57.13	4.66	100.00	16.74
		SD	0.10	2.05	85.90	24.36	5.37	0.14	0.00	0.83
Thirty				-						
Preheats	350°C	Mean	2.99	0.50	6.82	345.56	55.55	4.90	100.00	17.80
		SD	0.18	0.42	3.99	57.95	1.05	0.08	0.00	2.36

# of	Sub.											
preheats	Temp		Sq[μm]	Ssk	Sku	Sp[μm]	Sv[μm]	Sz[μm]	Sa[μm]	Sk[μm]	Spk[μm]	Svk[μm]
-												
No Preheat	32°C	Mean	2.179	0.817	3.615	5.459	9.950	15.409	1.724	5.163	0.863	3.182
		SD	0.201	0.161	0.230	0.703	1.384	1.882	0.169	0.674	0.167	0.170
-												
Two Preheats	115°C	Mean	2.585	0.120	2.715	7.707	9.070	16.777	2.086	6.594	1.824	2.634
		SD	0.062	0.169	0.125	1.263	0.329	0.941	0.055	0.263	0.373	0.612
-												
Thirty Preheats	350°C	Mean	2.530	0.113	2.733	7.629	9.080	16.709	2.038	6.630	1.888	2.512
		SD	0.117	0.073	0.388	0.680	1.543	2.187	0.050	0.498	0.576	0.830

Sub.									
	Temp		SMr1[%]	SMr2[%]	Sxp[μm]	Vvv[μm³/μm²]	Sal[μm]	Str	Sdr
No Preheat	32°C	Mean	5.445	85.065	4.216	0.335	21.309	0.707	32.466
		SD	0.714	1.962	0.547	0.011	2.598	0.309	1.143
Two Preheats	115°C	Mean	10.190	89.044	5.564	0.307	25.794	0.775	36.978
		SD	1.950	2.448	0.287	0.047	1.423	0.040	0.455
Thirty									
Preheats	350°C	Mean	9.491	90.143	5.435	0.296	28.833	0.822	36.080
		SD	2.105	2.683	0.287	0.057	3.342	0.113	0.467

Spray parameters for plasma spray coating at each corresponding substrate temperature are shown in Table 14. Particle temperature and velocity were measured with a Tecnar Accuraspray system for each run directly prior to deposition.

Table 14. Spraying parameters for coating of substrates with varied preheat temperature

Spraying Parameter	32° C	115° C	350° C
Spray Gun	F4MB-XL (Oerlikon Metco)		
Current, A	506	505	505
Voltage, V	74.5	74.7	74.7
Argon gas flow, NLPM	57	57	57
Hydrogen gas flow, NLPM	9	9	9
Powder gas flow, NLPM	7	7	7
Powder feed rate, g/min	29	29	29
Traverse rate, mm/s	500	500	500
Standoff distance, mm	130	130	130
Particle temperature, °C	2342	2352	2359
Particle velocity, m/s	349	347	344

3.4.2 Results and Discussion

3.4.2.1 Si Coating Adhesion and Mean Sprayed Si Particle Size

Table 11 provides the spray parameters including measured particle temperature and velocity for spraying of Si with varied mean particle size. In-flight particle temperature increased and particle velocity decreased with increasing mean particle size. The trend of decreasing particle velocity with increasing particle size corresponds to what Niu et al. [17] observed when plasma spraying Si, however they observed decrease in particle temperature with increasing size, the opposite of what was observed here. These differences in in-flight particle properties likely played a role in deposited coating adhesion strength.

Figure 79 shows the average adhesion strength for each mean Si particle size. The lowest value of coating adhesion strength was seen for the smallest mean particle size of 56 μm while the highest adhesion strength was seen for the medium mean size of 73 μm , and in between was the largest mean particle size of 94 μm . Samples coated with as received, unsieved Si powder with mean particle size of 71.3 μm had an average adhesion strength of 6537 psi, while an

average adhesion strength of 6428 psi was seen for samples coated with sieved Si powder and mean particle size of 73 μm . These results indicate that mean Si powder particle size affects the adhesion strength of the APS coating. There are a few things that likely contribute to this observed effect. The high velocity and lower temperature of the smallest particles, as listed in Table 11, may have resulted in greater breakup and/or splashing of droplets upon impacting the substrate, degrading the quality of the substrate/coating contact and adhesion. The high temperature of the large particles possibly resulted in increased spreading or undesirable breakup of splats upon impact, reducing coating adhesion strength compared to the medium sized particles, but didn't negatively impact coating formation as much as the high velocity and lower temperature droplets of the smallest mean particle size powder. Also, as pointed out by Kromer et al. [52], the size of the sprayed particles relative to the nature and size of the surface asperities and topography likely play a role in the mechanical interlocking of the coating with the substrate.

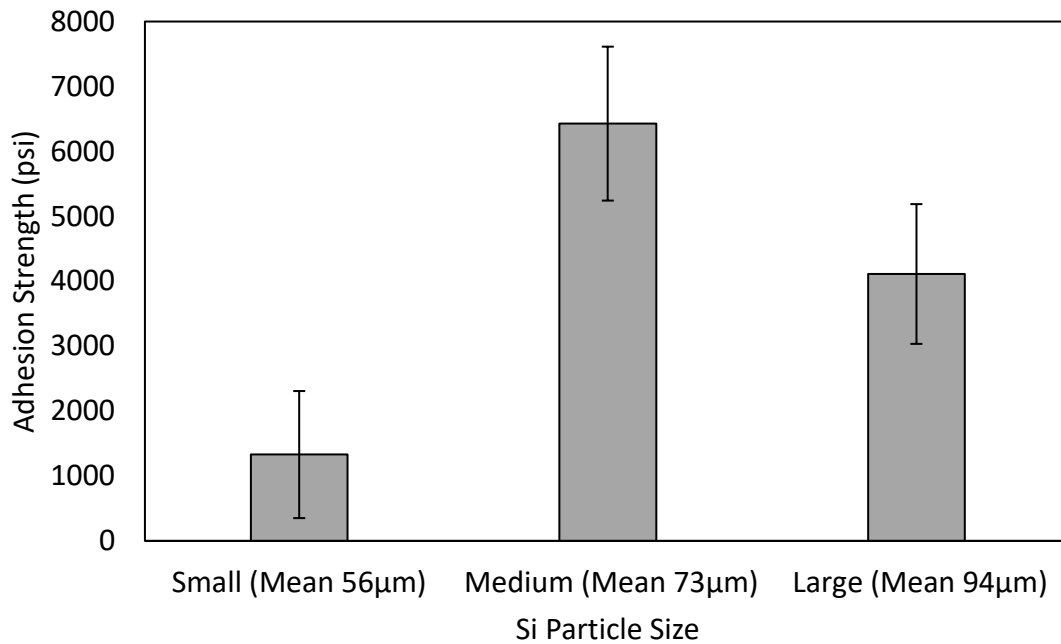


Figure 79. Average adhesion strength with varying mean Si particle size

3.4.2.2 Si Coating Adhesion and Substrate Preheat Temperature

The average adhesion strength (expressed as Max Stress in psi) for each sample set with different preheat temperature is plotted in Figure 80, with the error bars representing plus or minus one standard deviation. The mean coating adhesion strength for the samples preheated to $\sim 115^{\circ}\text{C}$ via two torch passes was seen to be more than triple that of the samples not preheated

prior to coating. Further increasing substrate temperature to $\sim 350^{\circ}\text{C}$ via thirty passes resulted in an adhesion strength nearly double that of the lower temperature substrate and a significant six-fold increase in adhesion strength compared to the ambient temperature substrate.

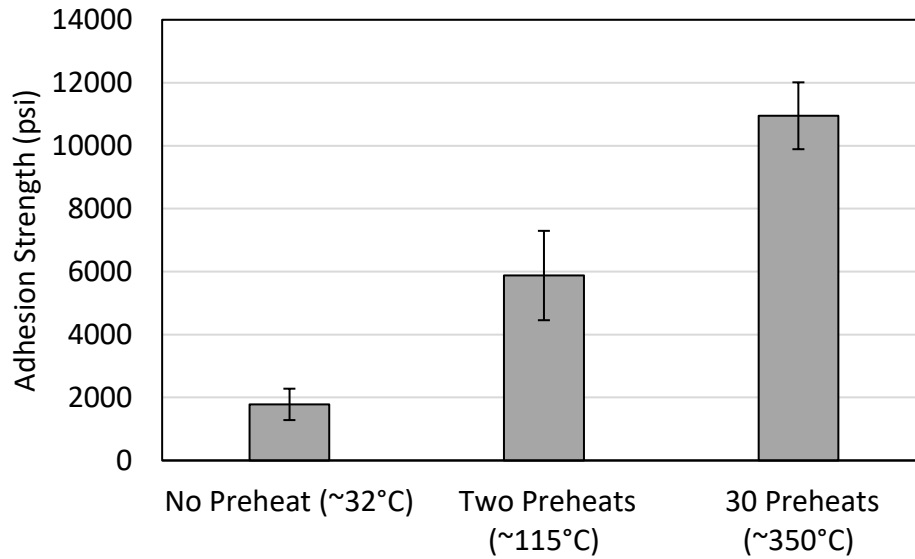


Figure 80. Average adhesion strength with varying level of preheating

Plotted in Figure 81 is the coating adhesion strength as a function of the average grit blasting induced surface roughness, R_a , and substrate temperature. The samples at 147°C were a large batch of samples all preheated via two passes of the plasma torch and coated simultaneously. Though the samples preheated to 115°C were also preheated with two passes of the torch, the larger batch of samples required a longer total preheat time which resulted in greater elevation of the temperature of the structure holding the samples and thus kept the samples at elevated temperature until spraying began. The main takeaway from Figure 81 is the effect of preheat temperature on adhesion strength despite surface roughness. When average R_a was $2.22\text{ }\mu\text{m}$ but samples were not preheated, the average adhesion strength was the same as that of preheated substrates with an R_a of $0.55\text{ }\mu\text{m}$ and lower, including smooth polished samples. Upon increasing substrate temperature to 115°C , adhesion strength was seen to be very close to samples with similar values of R_a in the large batch. Further increase to 350°C resulted in the largest observed value of adhesion strength, 10,950 psi, which was close to the strength of the epoxy used to test the adhesion strength of coatings, 11,586 psi. These results have made clear

that both surface roughness and substrate preheat temperature are important for ensuring strong coating adhesion. Even if a surface is sufficiently rough, if it is not sufficiently preheated, a plasma sprayed coating will not strongly adhere to it.

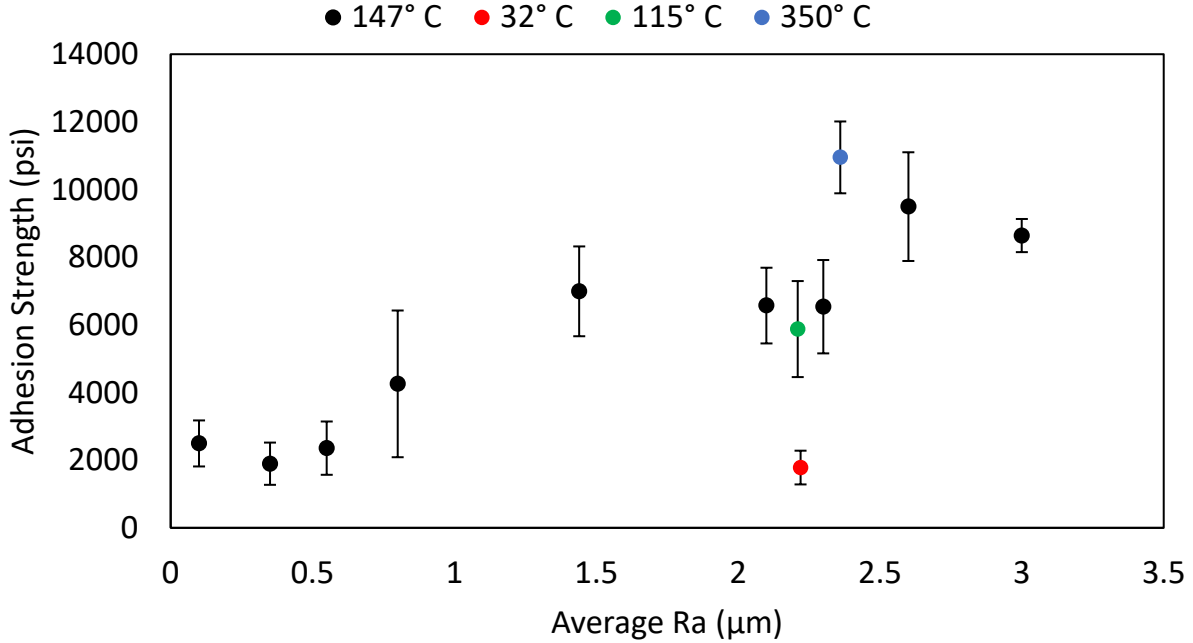


Figure 81. Coating adhesion strength as a function R_a and substrate preheat temperature

Based on the experimental results, an empirical relation was developed for the plasma sprayed Si coating adhesion strength on rb SiC as a function of substrate temperature, Si mean particle size and roughness parameters. The adhesion strength can be predicted by the following equation:

$$\text{Adhesion Strength} = -10556.66 + 109.37(\text{Mean Particle Size}) - 11.82((\text{Mean Particle Size})^2) + 35.72(\text{Substrate Temperature}) - 0.082((\text{Substrate Temperature})^2) + 139.33(\text{Sdr}) + 6.07(\text{Sdr}^2) - 272(\text{Sk}^2)$$

The statistical model for adhesion strength has an R^2 value of 0.95 and an adjusted R^2 value of 0.91, indicating that the model agrees well with the actual values and 95% of the adhesion strength value can be predicted using the above relation between coating adhesion strength, roughness parameters, substrate temperature and Si mean particle size based on R^2 . The model p value of <0.0001, as well as the p values for each factor all being <0.05, indicates the high significance of the model and the factors.

A model for adhesion strength as a function of only substrate temperature and the commonly measured surface roughness parameter R_a was also developed:

$$\text{Adhesion Strength} = -1598.18 + 1711.58(R_a) - 918.36(R_a^2) + 37.14(\text{Substrate Temperature}) - 0.11(\text{Substrate Temperature}^2)$$

The R^2 value of 0.91, adjusted R^2 value of 0.86 and model p value of 0.0003 reflect the significance of the model and agreement between predicted and actual responses. 91% of the adhesion strength value can be predicted using the above relation between coating adhesion strength, R_a and substrate temperature based on R^2 . All factors were found to be significant with p values <0.05.

Surface Chemistry

Change in surface chemistry with varying preheat treatment was characterized using XPS. Table 15 shows the surface composition of samples preheated to different temperatures by varying the number of preheat passes of the plasma torch. The change in atomic % of O, Si and C with preheat temperature is plotted along with the coating adhesion strength in Figure 82. The amount of oxygen present near the surface increased substantially with increasing temperature, while the amount of carbon and silicon decreased. As an oxide layer readily forms on SiC even at room temperature, the observed change in surface composition likely corresponds to growth of a silica layer with increasing number of preheat passes and substrate temperature. This silica layer, along with other factors like evaporation of adsorbates and adsorbed gases as suggested by other researchers [34-36, 42-44], may affect the interaction and contact between plasma sprayed droplets and the substrate surface.

Table 15. Surface composition of rb SiC substrates with varying preheat treatment

Preheat Temp, °C	# of preheat passes	O, atomic %	Si, atomic %	C, atomic %
32	0	25.4	36	38.6
115	2	37.2	34.1	28.7
350	30	46	31.7	22.3

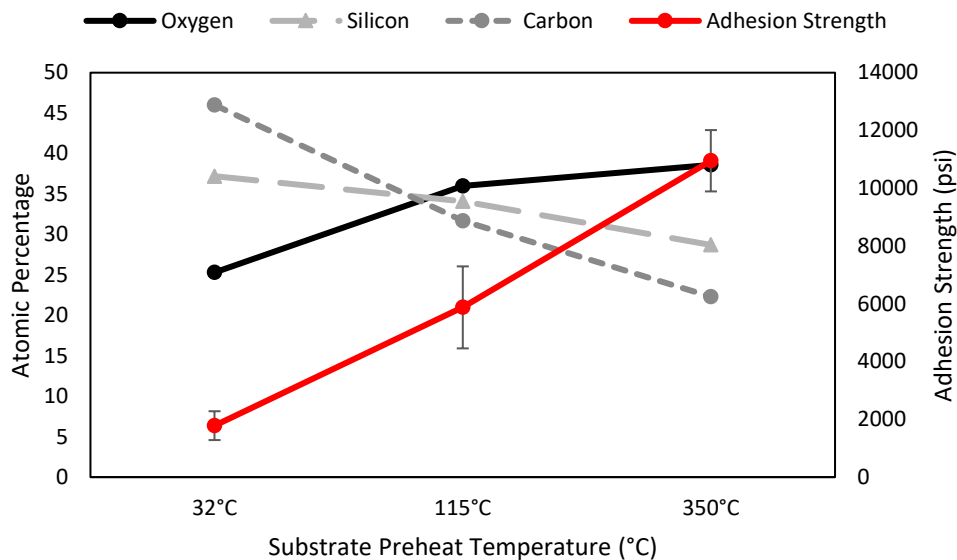


Figure 82. Change in atomic percentage of O, Si, C at the surface and corresponding coating adhesion with substrate preheat temperature

3.4.3 Si Single Splats on RB SiC

Single splats of Si were also sprayed onto rb SiC substrates with a range of roughness's after varying levels of preheating to observe the effects of preheating on splat behavior. Single splats were obtained by traversing the plasma spray gun very rapidly across the substrates and otherwise spraying normally. The splat behavior on smooth polished rb SiC surfaces with varying amounts of preheating was of primary interest and polished samples were included at every level of preheating. Pure silicon spraying powder was sieved to a narrow size distribution with mean particle size of 56 μm and standard deviation of 4 μm . As before, prior to depositing silicon splats via APS, the plasma torch without powder was traversed across the substrates with a varying number of total passes to generate different substrate temperatures and levels of preheating. Substrate temperature was monitored via a K type thermocouple placed on the back of a single sample and the time between the end of preheating and spraying was approximately 30 seconds. The temperature of the other samples being sprayed simultaneously was assumed to be the same as the material, each step of the process and time in between was consistent. The number of preheat passes prior to splat deposition and corresponding measured substrate temperature upon splat impact is shown in Table 16.

Table 16. Number of preheat passes and corresponding substrate temperature during single splat deposition

Number of preheat passes	Value
0	28° C
2	72° C
6	140° C
30	256° C

Smooth polished substrates on which splats were deposited had an average roughness S_a between 0.04 and 0.08 μm . Images and 3D surface maps of the polished surface are shown in Figure 83.

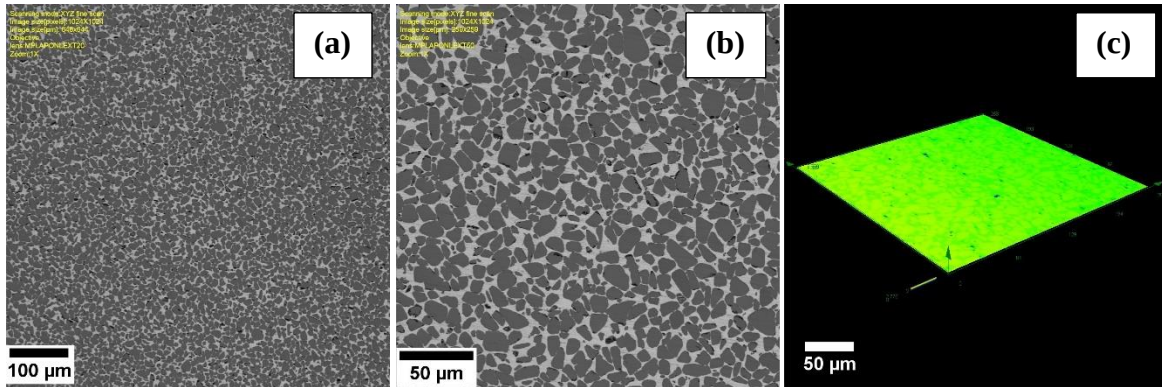


Figure 83. Laser scanning microscope images of polished rb SiC surface at a) 20x magnification, b) 50x magnification and c) 3D surface map

Spray parameters for single splat deposition for each corresponding substrate temperature are shown in Table 17. Particle temperature and velocity were measured with a Tecnar Accuraspray system for each run directly prior to deposition.

Table 17. Spray parameters for single splat deposition at each corresponding substrate temperature

Spraying Parameter	28° C	72° C	140° C	256° C
Spray Gun	F4MB-XL (Oerlikon Metco)			
Current, A	507	507	508	507
Voltage, V	74.2	72.6	76	74.5
Argon gas flow, NLPM	57	57	57	57
Hydrogen gas flow, NLPM	9	9	9	9
Powder gas flow, NLPM	7	7	7	7
Powder feed rate, g/min	29	29	29	29
Traverse rate, mm/s	500	500	500	500
Standoff distance, mm	130	130	130	130
Particle temperature, °C	2371	2339	2339	2336
Particle velocity, m/s	354	356	356	353

3.4.3.1 Effects of Substrate Preheating on Si Splat Formation

Figure 84 shows images of splats on polished surfaces with varying amounts of substrate preheating. It can be seen that fewer splats remained adhered to the surface of the colder substrates, Figure 84a and b, with a very small amount adhering to the surface which was not preheated, Figure 84a. The splats on substrates preheated to 140 and 256° C shown in Figure 84c and d were more numerous and showed little to no signs of splat removal after impact, unlike that of splats on the room temperature and 72° C substrates. Figure 85 shows higher magnification images of splats on the room temperature (Figure 85a and b) and 72° C (Figure 85c and d) substrates. It is clear in Figure 85a and b that primarily only the outer rims and

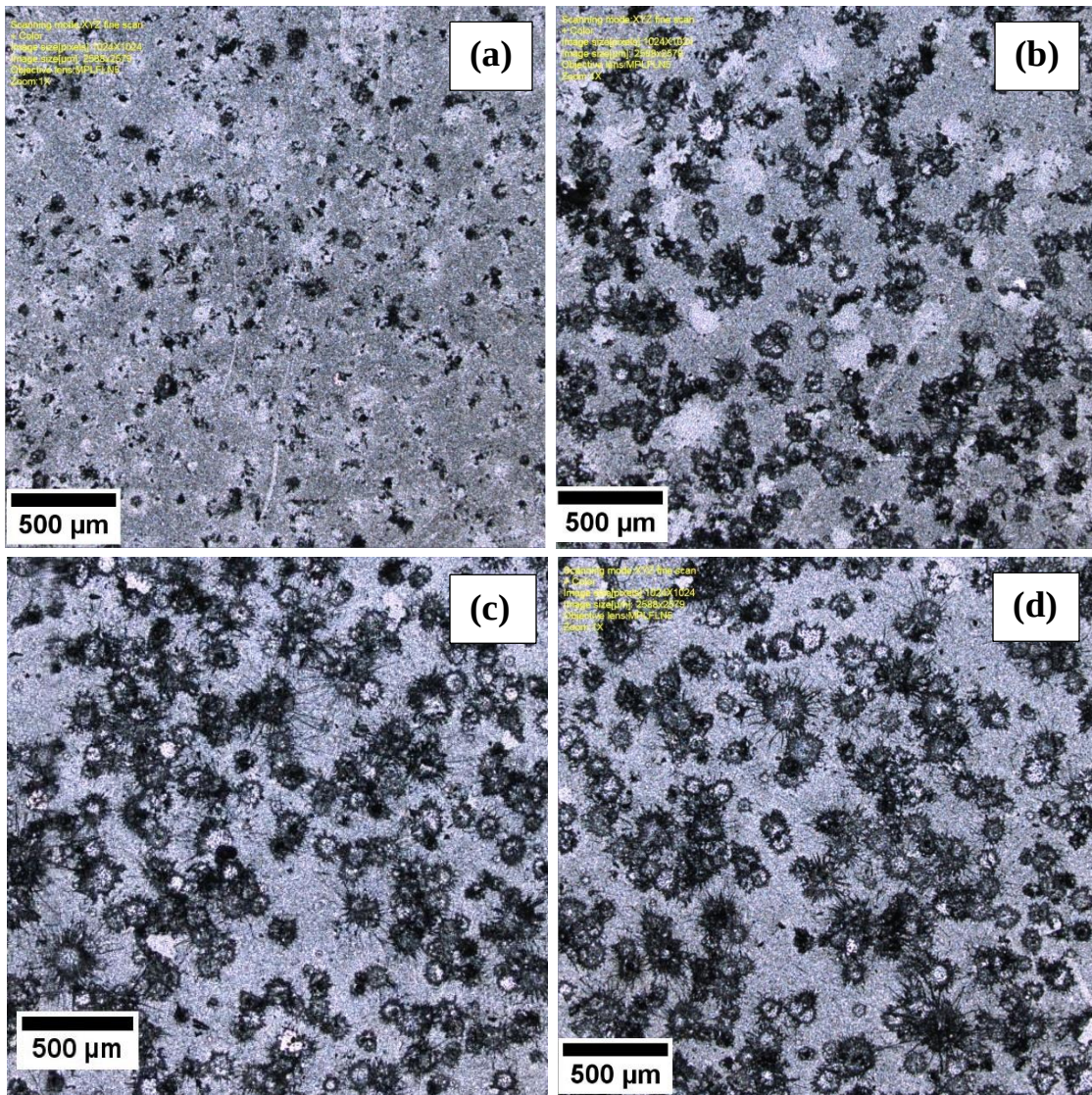


Figure 84. Laser scanning microscope images of Si splats sprayed on smooth polished rb SiC with varying preheat temperature: a) 28°C (RT), b) 72°C, c) 140°C and d) 256°C

remnants of deposited splats remained on the surface of the substrate which was not preheated. In Figure 85c and d, a greater amount of well-defined splats remained adhered to the preheated surface, however there are clearly impact marks and remnants of splats that did not stay adhered to the surface which is not observed for the higher temperature substrates in Figure 84c and d.

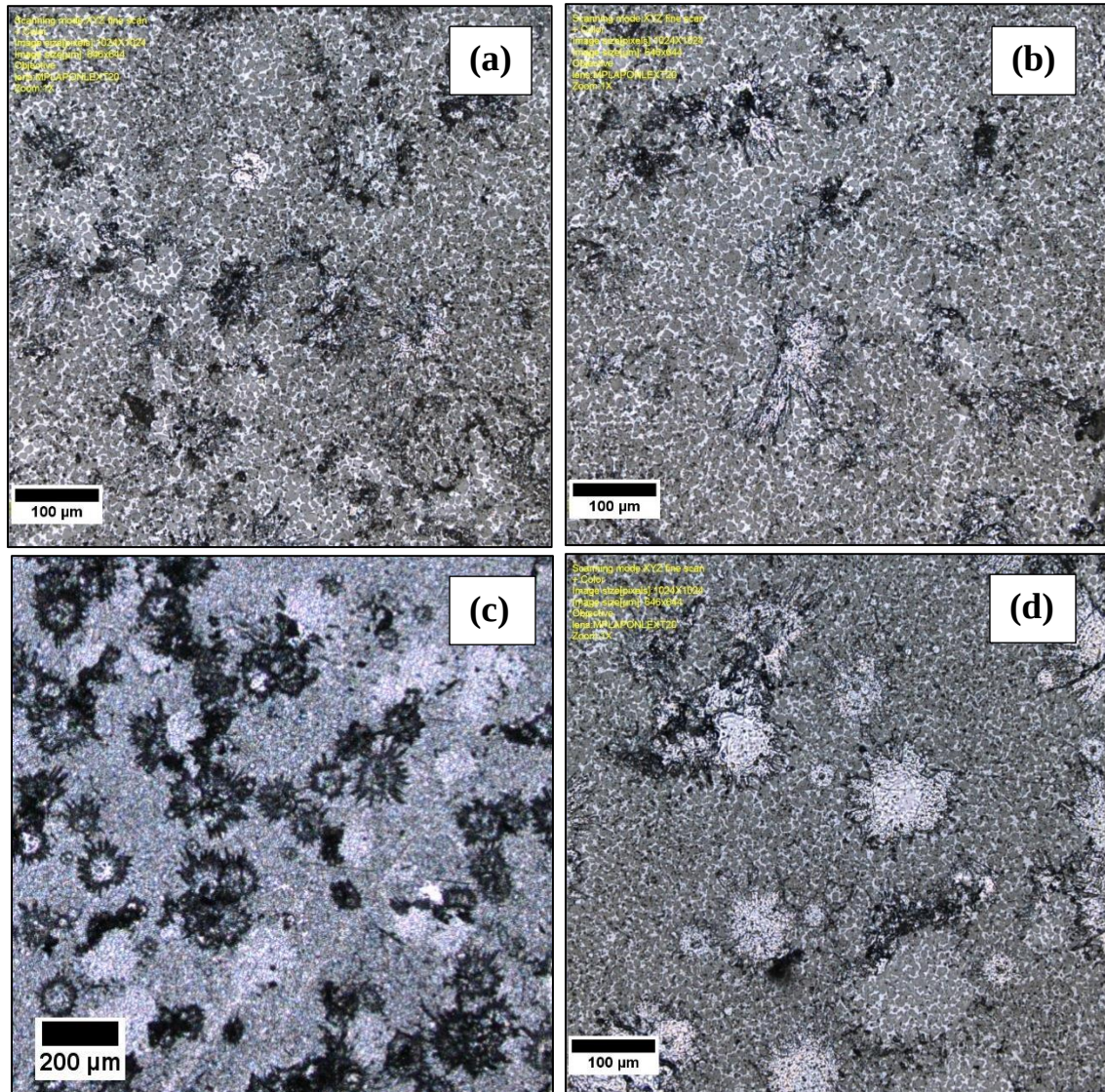


Figure 85. Laser scanning microscope images showing impact marks and scarcity of Si splats sprayed on smooth polished rb SiC at low preheat temperature: a,b) 28°C and c,d) 72°C

The splat morphology was also seen to be affected by the substrate preheat temperature. Figure 86 shows high magnification images of typical splats on substrates with varying temperature at deposition. As previously mentioned, few well defined splats remained on the room temperature substrate and only splat remnants primarily remained as seen in Figure 86a, so splat morphology could not be evaluated for these samples because so few stayed adhered. Figure 86b shows a typical splat on the smooth substrate preheated to 72° C which has a flower-like shape with fingers at the outer edges. A typical splat on the substrate preheated to 140° C is shown in Figure 86c and also has a flower-like shape but with smaller fingers compared to the splat in Figure 86b. On the hottest substrate, splats were typically more round and disc-shaped as seen in Figure 86d. These disc splats also had very thin fingers at the outer edges.

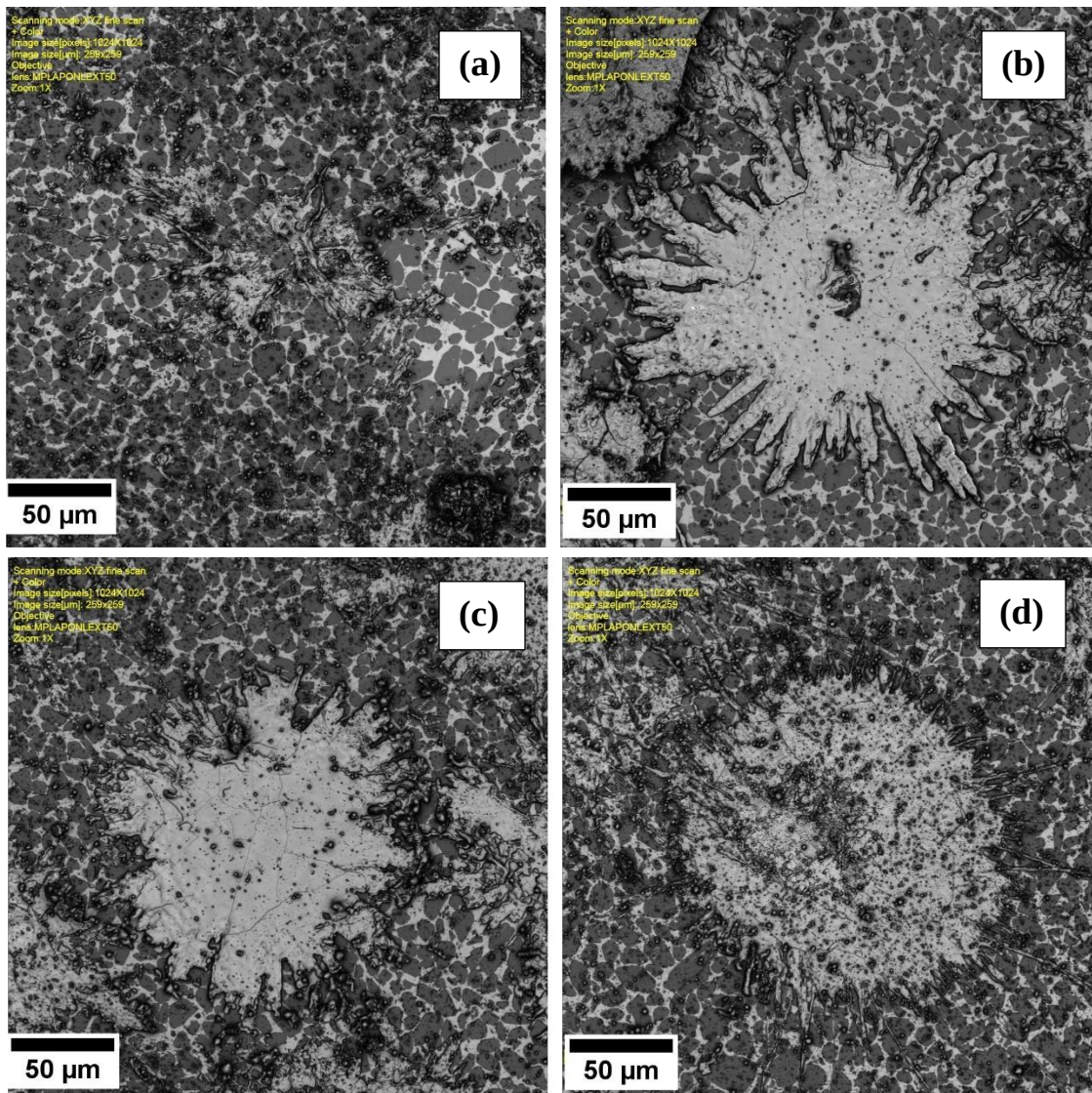


Figure 86. Laser scanning microscope images of Si splats sprayed on smooth polished rb SiC with varying preheat temperature: a) 28°C (RT), b) 72°C, c) 140°C and d) 256°C

Additional images of typical splats on the three substrates which were preheated before splat deposition are shown in Figure 87. ImageJ software was used to measure the circularity and solidity of splats. Plotted in Figure 88 is the average splat circularity and solidity as a function of substrate preheat temperature, along with the average measured coating adhesion strength as a function of substrate temperature. Note that the preheat temperatures of the substrates which were coated for adhesion testing differed slightly from the temperatures of the substrates on which single splats were deposited, however the effect of preheat temperature on both splat morphology and adhesion strength can still be observed. The splat circularity, solidity and coating adhesion strength all increase with increasing substrate preheat temperature. The most substantial increase in splat circularity and solidity was seen when the substrate temperature was increased to its highest value from 140 to 256° C, and for adhesion strength when the temperature was increased from 115 to 350° C. These results correspond well with what has been observed for different substrate and coating materials when substrate preheat temperature is elevated. Increasing preheat temperature results in rounder, disc-like splats on smooth surfaces and greater coating to substrate adhesion.

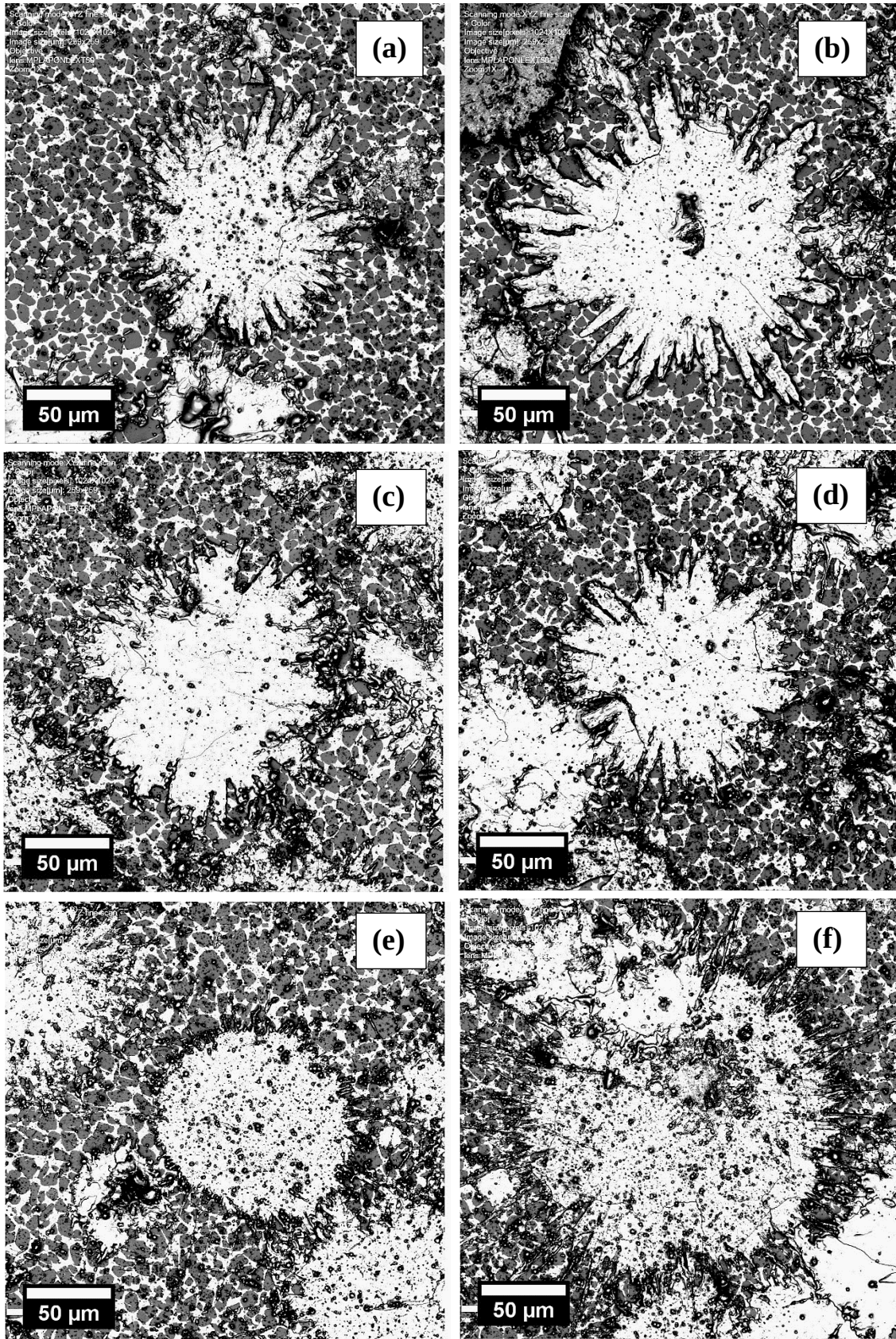


Figure 87. Laser scanning microscope images showing typical morphology of Si splats sprayed on smooth polished rb SiC with varying preheat temperature: a,b) 72°C, c,d) 140°C and e,f) 256°C

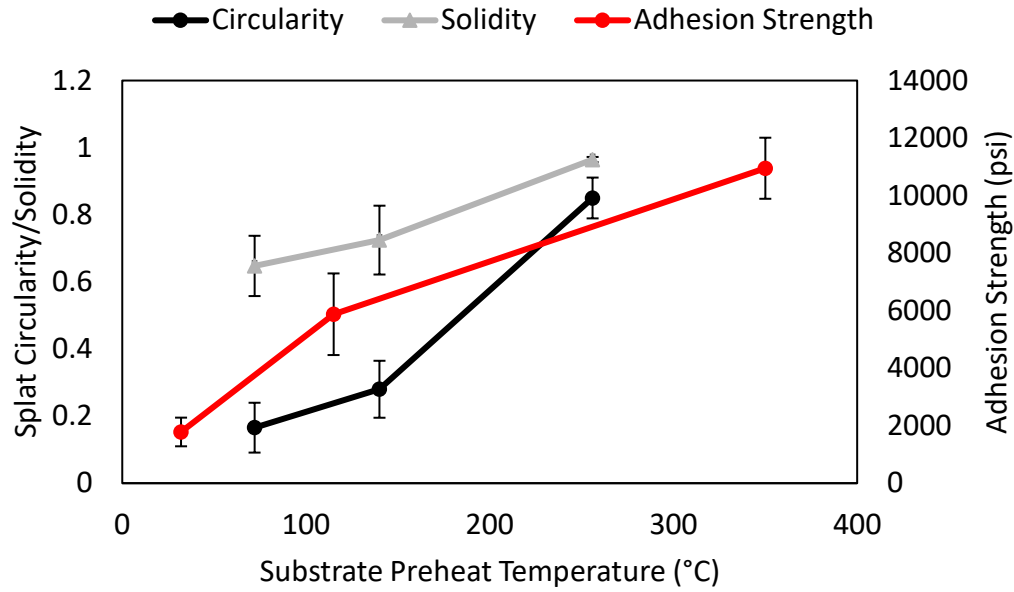


Figure 88. Change in splat circularity, solidity and coating adhesion with varying substrate preheat temperature

Silicon single splats were also deposited on grit blasted substrates at the same time as the smooth substrates with varied substrate preheat temperature. The roughened substrates were grit blasted using the same blast parameters in order to generate comparable surface roughness. Measured mean roughness parameters and standard deviations for the grit blasted substrates are shown for the corresponding preheat temperature in the Table 18.

Table 18. Mean 2D and 3D roughness parameters for grit blasted substrates on which Si single splats were deposited at varying substrate temperature

Substrate Temperature		Ra[μm]	Rsm[μm]	R Δ q[$^{\circ}$]	Sa[μm]	Sk[μm]	Sz[μm]	Sdr
28 $^{\circ}\text{C}$	(Mean)	2.74	368.14	55.02	2.50	8.15	21.82	44.43
	(SD)	0.17	26.80	0.66	0.29	1.31	1.06	1.60
72 $^{\circ}\text{C}$	(Mean)	2.81	352.02	55.97	2.49	7.93	29.97	45.90
	(SD)	0.10	29.47	0.48	0.29	1.44	13.00	2.18
140 $^{\circ}\text{C}$	(Mean)	2.74	383.05	59.06	2.31	7.61	20.71	42.68
	(SD)	0.10	48.77	6.64	0.24	0.87	1.89	1.52

Figure 89 shows Si splat morphology on grit blasted rb SiC with varying preheat temperature. The splats on the roughened surfaces are very much irregular relative to splats on a smooth polished surface and the spreading is seen to be impeded by the surface asperities. When comparing the splat on the room temperature substrate in Figure 89a to the splats on the heated substrates in Figure 89b and c, less splat breakup is observed for the substrates at elevated temperature. Similar to what was observed on the polished samples which weren't preheated, the splats deposited on a rough substrate at low temperature were less defined and were closer to splashed remnants of splats rather than well-defined splats.

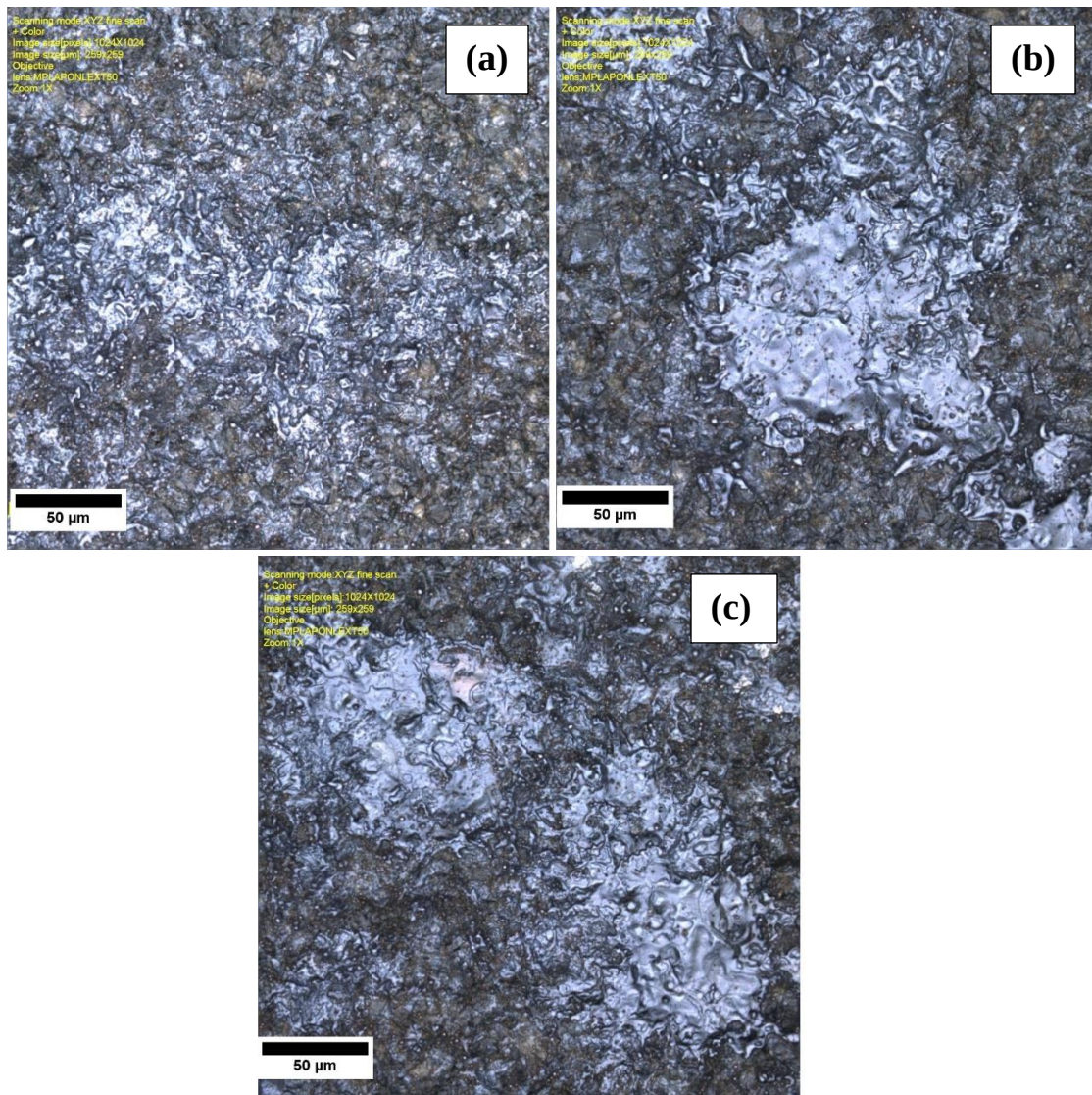


Figure 89. Laser scanning microscope images of Si splats sprayed on grit blasted rb SiC with varying preheat temperature: a) 28°C (RT), b) 72°C, c) 140°C

3.4.4 Summary

The effect of plasma sprayed Si mean particle size and substrate preheating on the Si coating adhesion strength on grit blasted rb SiC was evaluated. The effect of substrate temperature on Si single splat formation was also observed. Sieved Si mean particle size was seen to affect the measured in-flight particle velocity and temperature. A mean Si particle size of 73 μm , which is very close to the mean particle size of typically sprayed Si powder, resulted in the highest coating adhesion strength, while adhesion strength decreased for a particle size of 94 μm , and was lowest for the smallest mean particle size of 56 μm . Substrates were preheated via the plasma torch prior to spraying and substrate temperature measured with a thermocouple. Si coating adhesion strength increased significantly with increasing substrate preheat temperature despite all samples having very similar induced roughness. Increased coating adhesion strength on higher temperature substrates corresponded with higher oxide presence on the surface of samples only preheated via the same method as coated samples. A dramatic change in the number of Si splats which remained on the smooth rb SiC substrate surface as well as the shape of splats was observed with increasing substrate preheating. Splats became more round and disc-like on hotter substrates, while very few coherent splats remained on a substrate which was not preheated. Splats were also seen to be less coherent on room temperature, rough substrates than on rough substrates which were preheated.

3.5 Simulation of Plasma Sprayed Si Splats on Smooth and Textured SiC

Substantial amounts of research, both experimental and via simulation, has shown a relationship between the shape and formation of splats on the substrate surface during plasma spraying and the adhesion strength of the sprayed full coating. Splats are single, solidified droplets on the surface after impact during the coating process and are the building blocks for every thermally sprayed coating. The coating properties, including adhesion to the underlying substrate, depend on the nature and behavior of these individual splats, just like the properties of any material depend on the nature and structure of its atoms. Most studies of single splat behavior in atmospheric plasma spray coatings have observed splats on smooth polished

surfaces, often utilizing metallic materials. This is useful as it allows for observation of the role of many process parameters, such as material thermal properties, spray parameters and substrate temperature, in droplet impact, spreading and solidification. However, most surfaces coated via APS are not smooth. Surfaces are typically rough to allow for mechanical interlocking between the sprayed coating and substrate and increased adhesion strength, thus it is important to also understand the role that surface roughness, topography and texture play in the formation of splats.

Simulation software, SimDrop, was utilized to model the impact, spread and solidification of a silicon droplet on a silicon carbide surface during the plasma spray process. The primary goal was to gain a better understanding of how regularly patterned surface texture parameters affect splat spreading and final shape. In addition, the role of spray process parameters, such as droplet velocity and temperature, and substrate parameters, like temperature and material properties, in splat formation could be studied. Table 19 provides the physico-chemical properties of the Si droplet and SiC substrate used in the simulation.

Table 19. Physico-chemical properties of droplet and substrate used in simulations [94-98]

Material	Melting Point (°C)	Density (kg/m ³)	Thermal Conductivity (W/m-K)	Specific Heat (J/kg-K)	Kinematic Viscosity (m ² /s)	Latent Heat (J/kg)	Surface Tension (kg/s ²)
Si (l)	1412	2570	53	968	3.385x10 ⁻⁷	1.8x10 ⁶	0.82
Si (s)		2329	130	700			
SiC (substrate)	2730	3000	150	685			

Based on AccuraSpray measurements of in-flight particle properties from recent plasma spraying and splat deposition of Si, the particle input parameters in Table 20 were used as initial conditions to simulate and study the impact of plasma sprayed silicon on a smooth rb SiC substrate and compare with experimental observations. Si powder was sieved to a narrow particle size distribution with mean diameter close to 50µm for splat deposition.

Table 20. Initial droplet conditions for simulating Si droplet impact on smooth, preheated rb SiC during plasma spraying

Parameter	Value
Particle temperature (°C)	2350
Particle velocity (m/s)	354
Particle diameter (μm)	50
Substrate temperature, T _s , (°C)	256
Thermal contact resistance, TCR, (m ² K/W)	1x10 ⁻⁸

3.5.1 Effects of Spray Process Parameters on Si Splat Formation on Smooth SiC

The effect of thermal contact resistance (TCR) on simulated Si splat spreading and morphology on a smooth 256° C rb SiC substrate is shown in Figure 90. The degree of spreading is seen to increase with TCR as the rate of heat transfer decreases and slows solidification. In Figure 90a, when TCR is low, a round, disc-like splat is formed with splashed fingers around the periphery. The splashed fingers are a result of very quick solidification at the bottom of the splat where contact with the substrate is the greatest while the still liquid material flows over the solid creating fingers before solidifying. As TCR increases, splashing is reduced as droplets are able to spread more completely before solidifying. In Figure 90e, the outer edges of the splat solidified as the center remained liquid and continued to spread towards the edges, resulting in a halo or ring-like splat. At the highest simulated TCR of 6x10⁻⁸ m² K/W, Figure 90f, solidification is slowed so significantly that the droplet does not form a splat and the droplet spreads to the edges of the calculation domain.

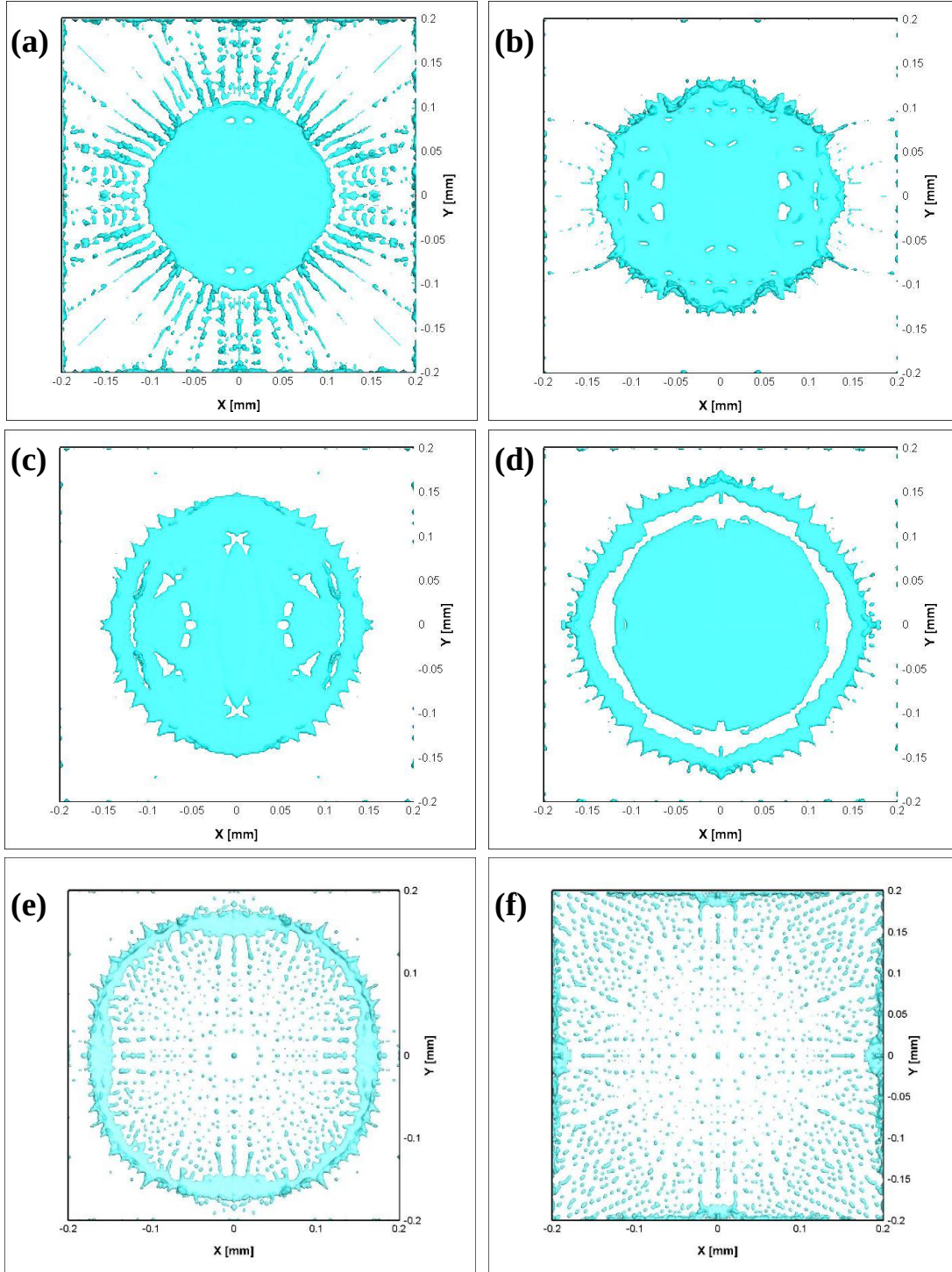


Figure 90. Simulated final splat morphology of Si impacting a smooth rb SiC substrate with increasing TCR: a) 1×10^{-9} , b) 6×10^{-9} , c) 8×10^{-9} , d) 1×10^{-8} , e) 1.3×10^{-8} , f) $6 \times 10^{-8} \text{ m}^2 \text{ K/W}$. Other impact parameters are given in Table 20

When simulation input parameters matched the measured experimental spray parameters for a substrate preheated to 256° C and thermal contact resistance was varied, the simulated splat shape on a smooth surface matched the experimentally observed splat shape when TCR was equal to 1×10^{-9} , as seen in Figure 91. Both the simulated and actual splats were round and disc-like with thin fingers surrounding the outer rim of the disc. The diameter of the core splats (excluding splashes and fingers) were found to be comparable between the simulated and experimental result as well, with the simulated and experimental splats from Figure 91 having diameters of 200 μm and 190 μm , respectively.

Figure 91. Final splat morphology of a) simulated Si impacting a smooth, 256°C rb SiC substrate with a TCR of 1×10^{-9} and b) a laser scanning microscope image of a Si splat plasma sprayed onto a smooth polished rb SiC substrate preheated to 256°C. Other impact parameters are given in Table 20

Parameter	Value
Particle temperature (°C)	2370
Particle velocity (m/s)	354
Particle diameter (μm)	50
Substrate temperature, T _s , (°C)	28
Thermal contact resistance, TCR, (m ² K/W)	0.8x10 ⁻⁸

Si splats were deposited onto room temperature rb SiC via plasma spraying and the measured spray parameters were used as input parameters to simulate the splat formation on a smooth surface. The input parameters used are given in Table 21. The effect of varying TCR on splat spreading and morphology on a 28° C substrate was observed and is shown in Figure 92. Once again, the amount of spreading before solidification is seen to increase with increasing TCR and splashed fingers around the periphery of the splat are formed for the lowest TCR. Due to the low substrate temperature, the droplet will solidify more quickly than a higher temperature substrate with the same TCR. When comparing Figure 92b to Figure 90e, a halo splat was formed on the high temperature substrate while a round, disc-like splat was formed on the room temperature substrate despite the same TCR.

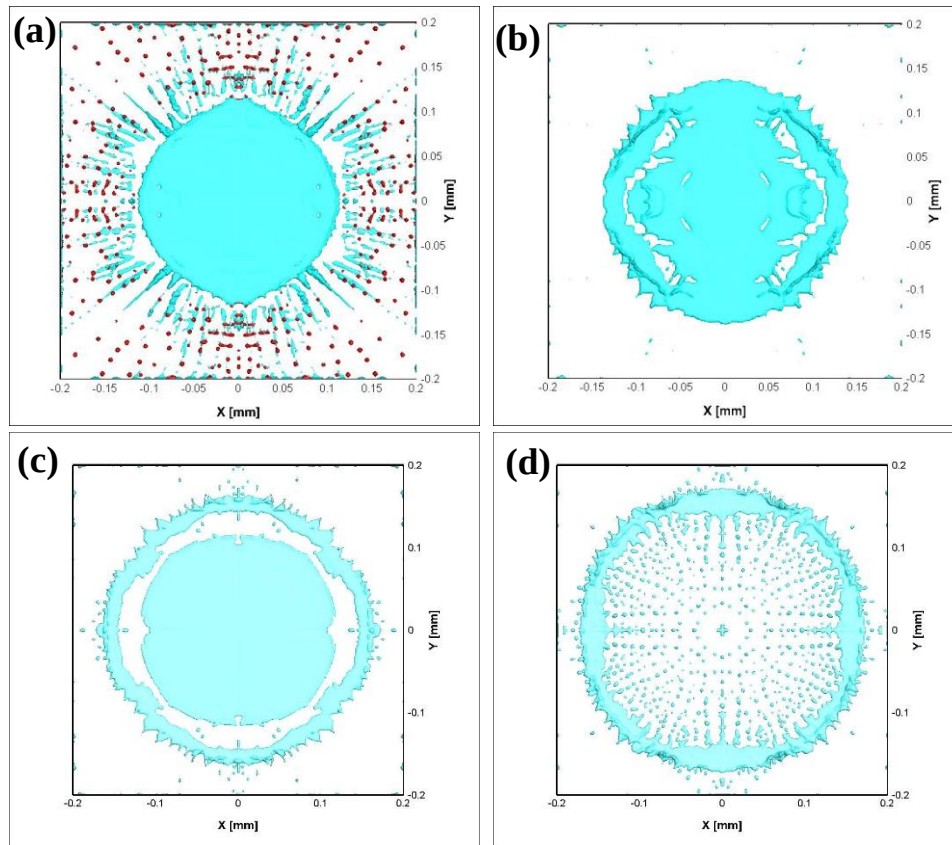


Figure 92. Simulated final splat morphology of Si impacting a smooth, 28°C rb SiC substrate with increasing TCR: a) 0.8×10^{-8} , b) 1.3×10^{-8} , c) 1.7×10^{-8} and d) $2 \times 10^{-8} \text{ m}^2\text{K/W}$

Figure 93 shows a laser scanning microscope image of typical splats deposited on a smooth polished rb SiC kept at room temperature during spraying. Substantial splat breakup is clearly seen with splat remnants and impact marks being the primary evidence of splat deposition. Highlighted in Figure 93b is a halo shaped splat consisting of just an outer ring of Si surrounding the exposed underlying substrate, which is typical of splats observed on this room temperature substrate. The splat spreading and morphology observed via simulation of Si impacting a smooth, 28°C substrate with TCR values of 2×10^{-8} and 3×10^{-8} m²K/W are shown in Figure 93c and d, respectively. Other impact parameters are given in Table 21. These simulated splats have a halo-like shape similar to the experimentally observed splat in Figure 93a and b due to the increased spreading before solidification because of the elevated TCR. On the contrary, the coherent disc-like splats observed on the preheated surface matched the simulated splats when the TCR was reduced to 1×10^{-9} m²K/W as seen in Figure 91. This indicates that the TCR is likely higher on a colder rb SiC substrate which results in greater splat spreading and the halo shaped splats, as well as lack of coherent splats. Additional higher magnification laser scanning microscope images of Si splats sprayed on the room temperature substrate are shown in Figure 94. Once again, the halo shape and distorted nature of the splats, similar to the simulation results in Figure 94c and d below, is observed indicating the significant effect of rb SiC substrate temperature and TCR on splat behavior.

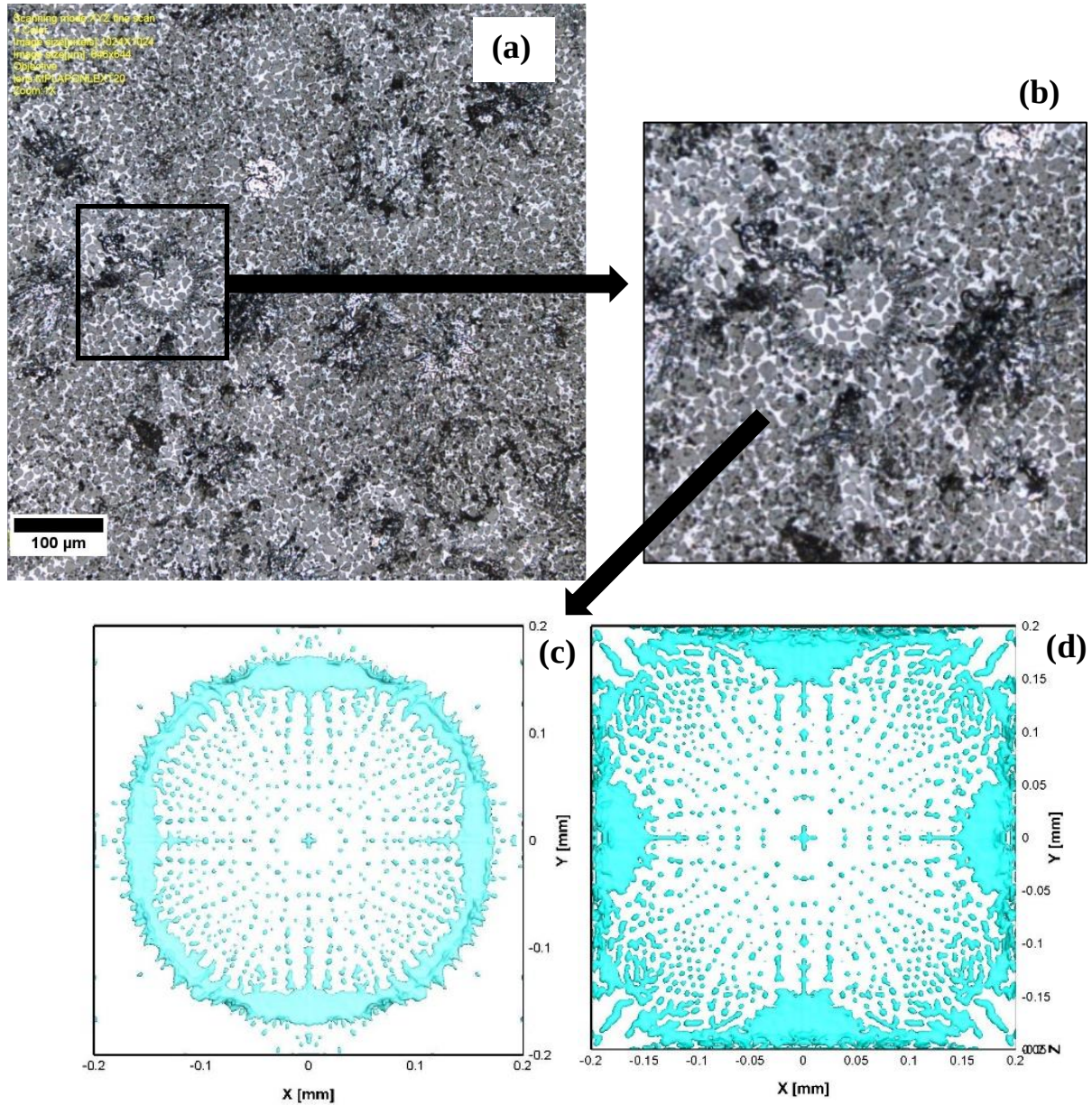


Figure 93. A laser scanning microscope image of Si plasma sprayed onto a smooth polished rb SiC substrate kept at room temperature (28°C) during spraying and b) close up image of halo shaped splat remnant. c) Final splat morphology of simulated Si impacting a smooth, 28°C rb SiC substrate with a TCR of 2×10^{-8} and d) $3 \times 10^{-8} \text{ m}^2\text{K/W}$. Other spray/impact parameters are given in Table 21

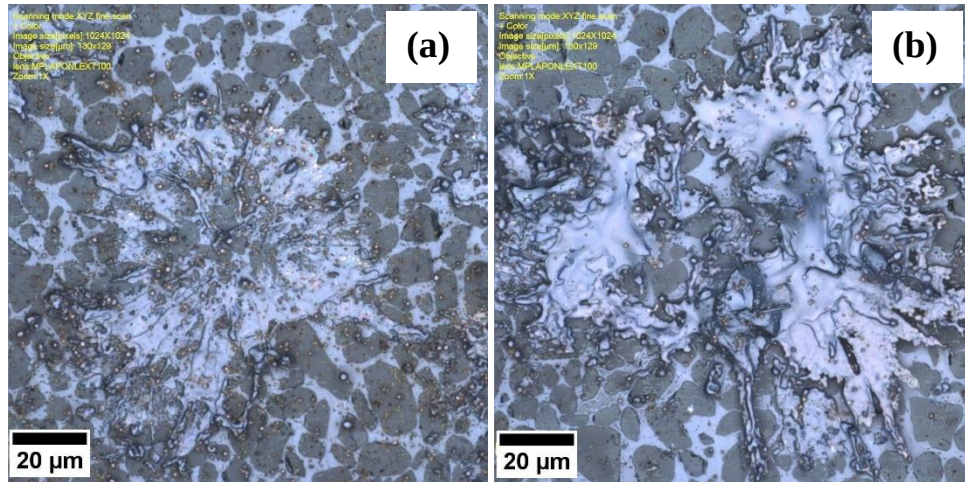


Figure 94. Laser scanning microscope images of Si plasma sprayed onto a smooth polished rb SiC substrate kept at room temperature (28°C) during spraying

Table 22. Initial droplet conditions for simulating Si droplet impact on smooth rb SiC during plasma spraying with varying substrate temperature

Parameter	Value
Particle temperature (°C)	2350
Particle velocity (m/s)	320
Particle diameter (μm)	50
Substrate temperature, T_s , (°C)	200
Thermal contact resistance, TCR, (m^2K/W)	1×10^{-8}

Initial conditions for studying the effects of spraying and substrate parameters on Si droplet impact, spreading and splat morphology on a smooth rb SiC substrate during plasma spraying are given in Table 22. These inputs were based on measured parameters during previous plasma spraying of silicon. Figure 95 shows the effect of increasing substrate temperature from room temperature up to 400 °C with TCR kept constant. Droplet spreading before solidification increased with increasing substrate temperature and splats solidified before complete spreading could occur for substrate temperatures of 25, 100 and 150 °C, as evidenced by the thicker regions near the splat edges seen in Figure 95a-c below. Splat shape changed substantially when substrate temperature was increased from 275 to 300 °C as it went from a disc-like splat with a surrounding ring of Si to a halo splat with all of the droplet spreading to the outer edges, shown in Figure 95e-g. The halo splat shape remained consistent with some

increased spreading when substrate temperature was increased from 300 to 400 °C (Figure 95g-i).

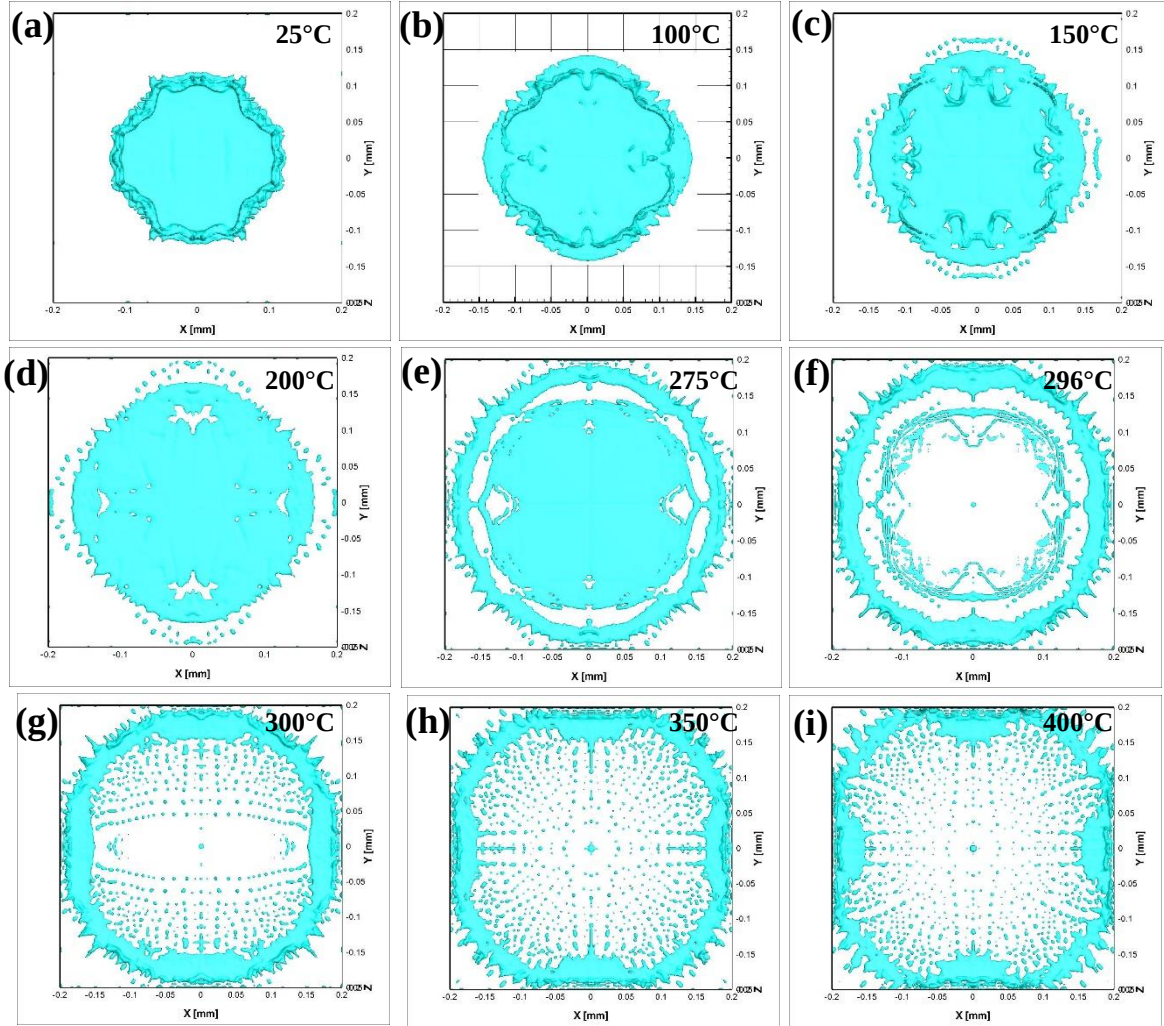


Figure 95. Simulated final splat morphology of Si impacting a smooth rb SiC substrate with increasing substrate temperature: a) 25°C, b) 100°C, c) 150°C, d) 200°C, e) 275°C, f) 296°C, g) 300°C, h) 350°C, i) 400°C. Other impact parameters are given in Table 22

The role of substrate thermal conductivity, k , along with substrate temperature in splat spreading and morphology can be observed in Figure 96. Splat spreading increases with increasing substrate temperature, as seen previously, and with decreasing k . Figure 96 makes evident that a different substrate temperature is required to achieve a certain splat morphology for substrates with different thermal conductivities, i.e. a different substrate material. A material

with a low thermal conductivity but held at a high temperature could result in too much slowing of solidification and overspreading of sprayed particles, as seen in Figure 96c. Alternatively, a substrate with a high thermal conductivity kept at low temperature could lead to very quick solidification upon impact, resultant splashing and inadequate spreading, as in Figure 96g

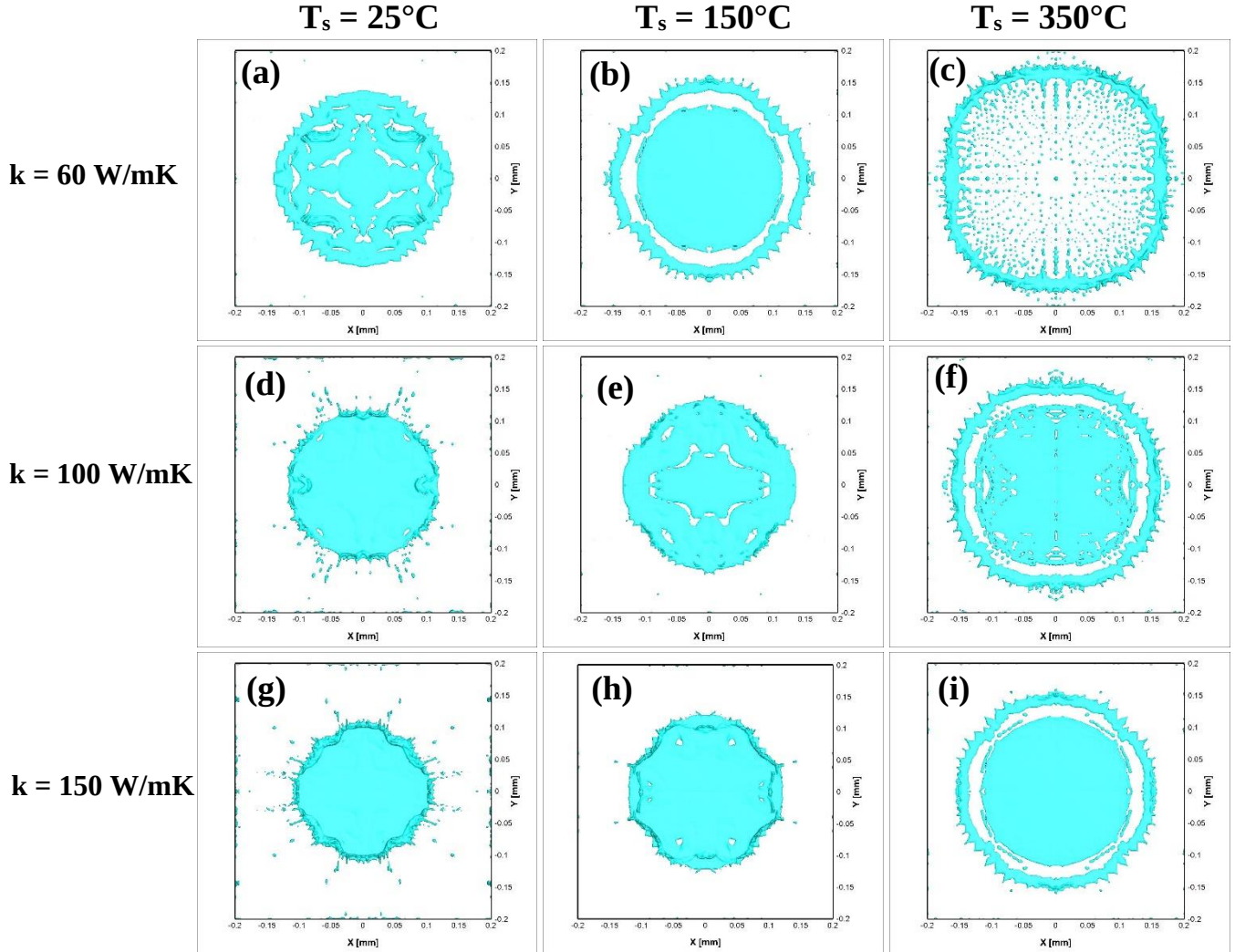


Figure 96. Simulated final splat morphology of Si impacting a smooth rb SiC substrate with varying substrate temperature, T_s , and thermal conductivity, k : a) 25°C, 60 W/mK, b) 150°C, 60 W/mK, c) 350°C, 60 W/mK, d) 25°C, 100 W/mK, e) 150°C, 100 W/mK, f) 350°C, 100 W/mK, g) 25°C, 150 W/mK, h) 150°C, 150 W/mK, i) 350°C, 150 W/mK. Other impact parameters are given in Table 22

Figure 97 shows the effect of increasing Si droplet velocity upon impact on a smooth, 200° C rb SiC substrate. The droplet spreads more before solidifying as velocity is increased. For the parameters used, given in Table 22, the solidified splats remain coherent and mostly disc-shaped at all velocities. Spreading and flattening of the droplet is halted by solidification before completion for droplet velocities of 70, 170 and 250 m/s, Figure 97a-c. At 400 m/s, Figure 97f below, a ring of solidified Si surrounds a solidified core splat due to the initial rapid outward spreading followed by rapid solidification of the outer ring and core material in contact with the surface.

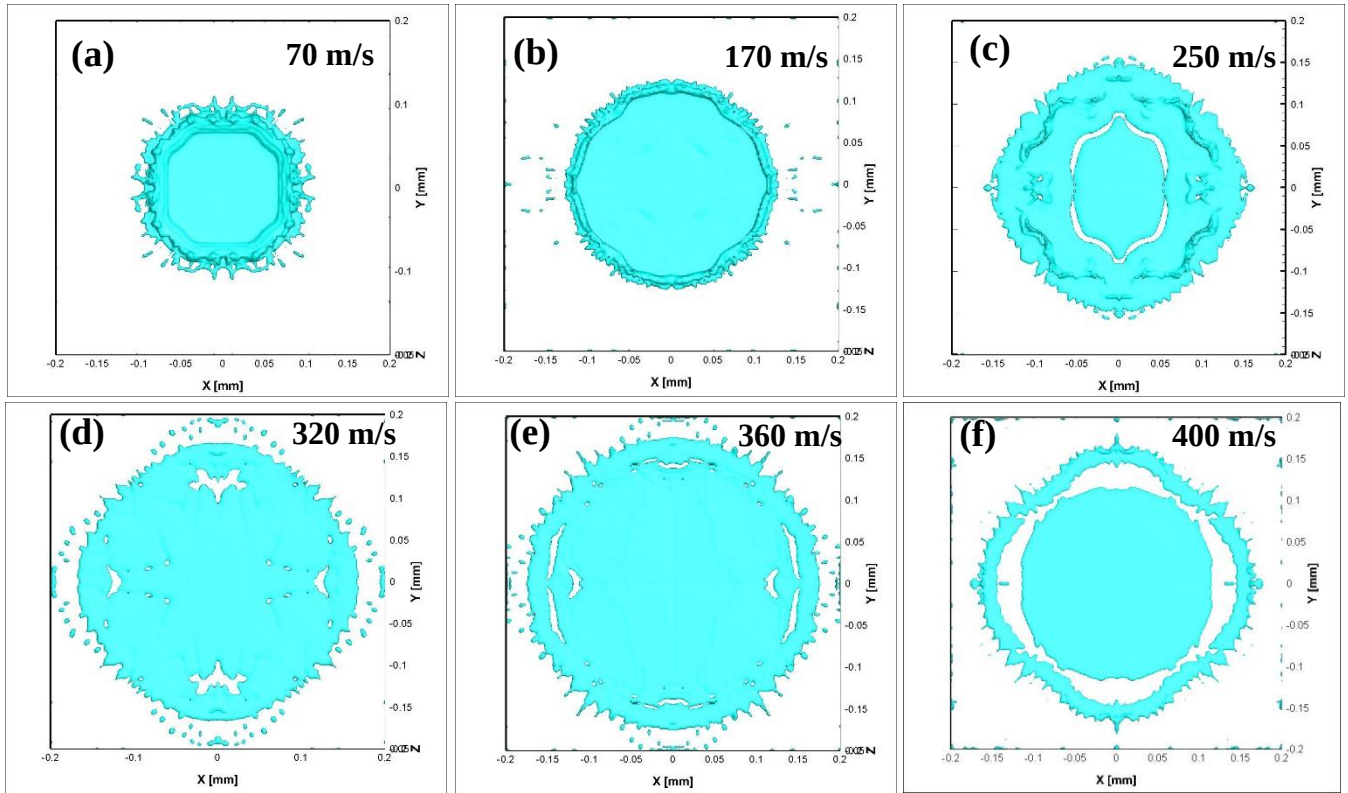


Figure 97. Simulated final splat morphology of Si impacting a smooth rb SiC substrate with increasing droplet velocity: a) 70 m/s, b) 170 m/s, c) 250 m/s, d) 320 m/s, e) 360 m/s, f) 400 m/s. Other impact parameters are given in Table 22

Changes in splat morphology with increasing droplet temperature are shown in 98. When the droplet is close to the melting point at 1500 and 1650 °C, Figure 98a and b, splats are very round with substantial splashing resulting from the flow of still molten Si into and over Si that

has solidified upon impact. At higher droplet temperatures, Figure 98c-f, splats are still round with the amount of spreading before solidification increasing with increasing droplet temperature. Small splashed droplets are observed outside the periphery of the splat when droplet temperature is 2350 and 2600 °C as seen in Figure 98e and f.

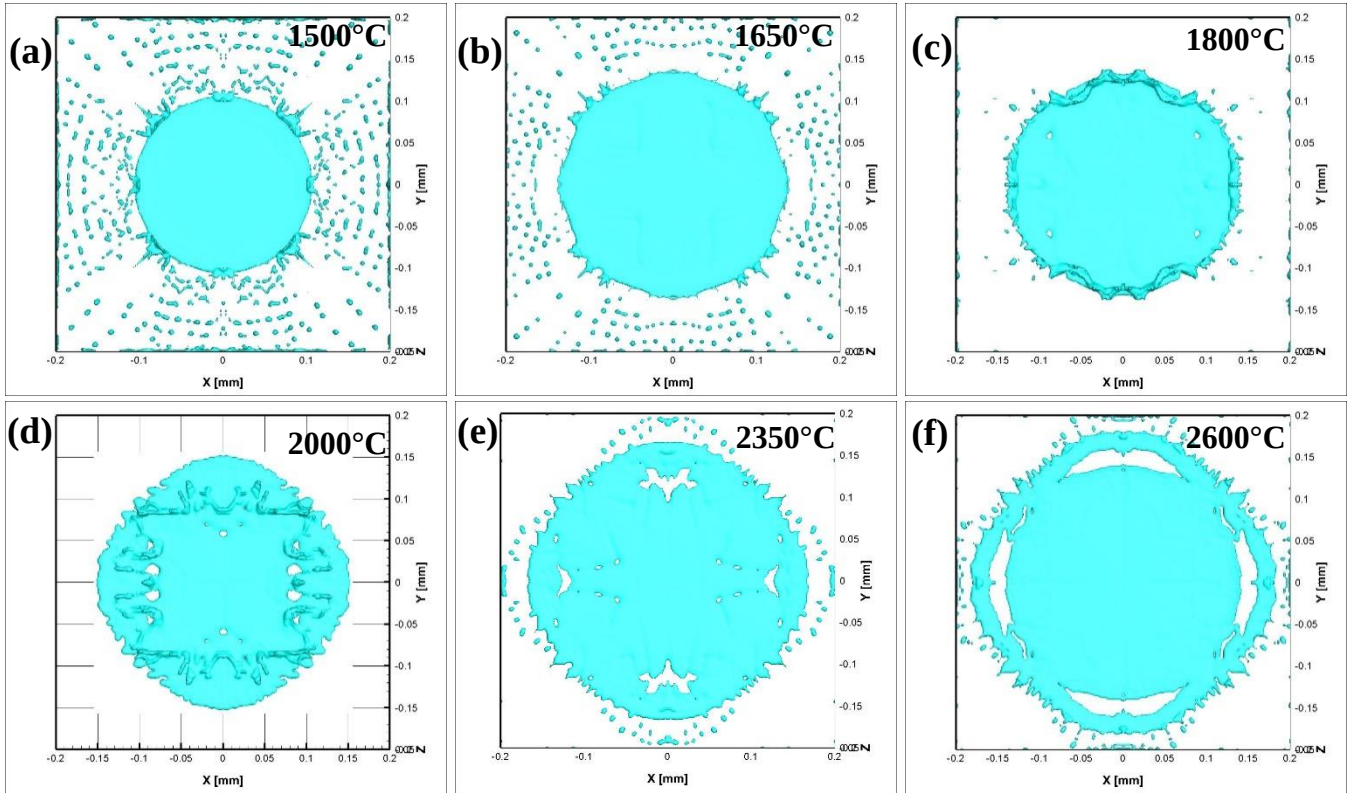


Figure 98. Simulated final splat morphology of Si impacting a smooth rb SiC substrate with increasing droplet temperature: a) 1500°C, b) 1650°C, c) 1800°C, d) 2000°C, e) 2350°C, f) 2600°C. Other impact parameters are given in Table 22

3.5.2 Simulated Si Splat Formation on Textured Surfaces

Though observing coating behavior when sprayed onto smooth surfaces is useful for characterizing the plasma spraying and splat formation processes, substrates that are plasma spray coated are not usually smooth in practical application. It is important to consider substrate surfaces which are not smooth and have some form of texture. Si droplet impact and splat formation on a pattern textured rb SiC substrate was simulated to study the effects of surface

texture parameters on splat spreading and morphology. The simulated texture is made up of posts or peaks on the surface with predefined height, spacing and width as seen in Figure 99b. The defined texture/peak dimensions are constant across the simulated surface and a 3D view of a simulated surface texture is shown in Figure 99a. Though plasma spray coated surfaces typically have a more random surface roughness from grit blasting, machining or other surface preparation, surfaces can be and were treated with a pulsed laser technique to generate a patterned texture similar to the simulated surface texture. The initial conditions used to simulate Si droplet impact on textured rb SiC during plasma spraying are given in Table 23.

Table 23. Initial droplet conditions for simulating Si droplet impact on textured rb SiC during plasma spraying with varying texture parameters

Parameter	Value
Particle temperature (°C)	2350
Particle velocity (m/s)	320
Particle diameter (μm)	50
Substrate temperature, T_s , (°C)	200
Thermal contact resistance, TCR, ($\text{m}^2\text{K/W}$)	5×10^{-6}
Texture peak height (μm)	5
Texture peak spacing (μm)	5
Texture peak width (μm)	10

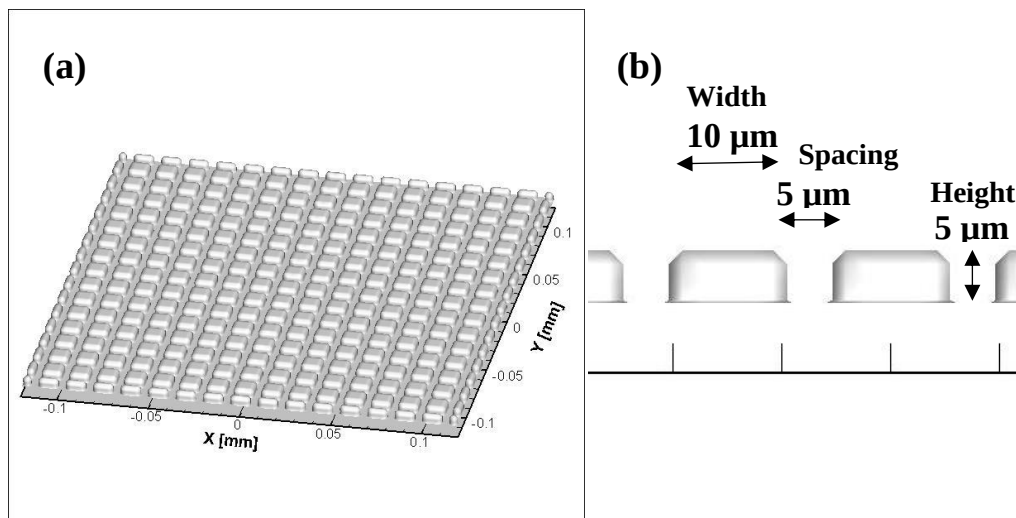


Figure 99. Schematic of simulated surface texture showing a) full 3D textured surface and b) measurements of peak height, spacing and width for the initial texture pattern conditions

Figure 100 shows the evolution of splat behavior with increasing texture peak spacing. Compared to impact on a smooth surface, the surface texture causes a large amount of splashing as well as the generation of splat fingers and more distorted splat shape. However, especially considering the TCR, the texture impedes the droplet spreading. Compared to Figure 90e and f in which similar impact parameters were utilized for impact on a smooth surface, the substantial spreading at higher values of TCR on the smooth surface was greatly reduced by the surface texture. It is clear, though, from Figure below that increasing the texture spacing results in greater spreading and splashing of the impending droplet as the liquid is able to more easily flow between the texture and impact peaks as it spreads, generating splashes and less compact splats. The smaller peak spacing illustrated in Figure 100a-c limits the droplet flow in between the texture and impact peaks and results in still coherent splats with fingers around the periphery. However, it is

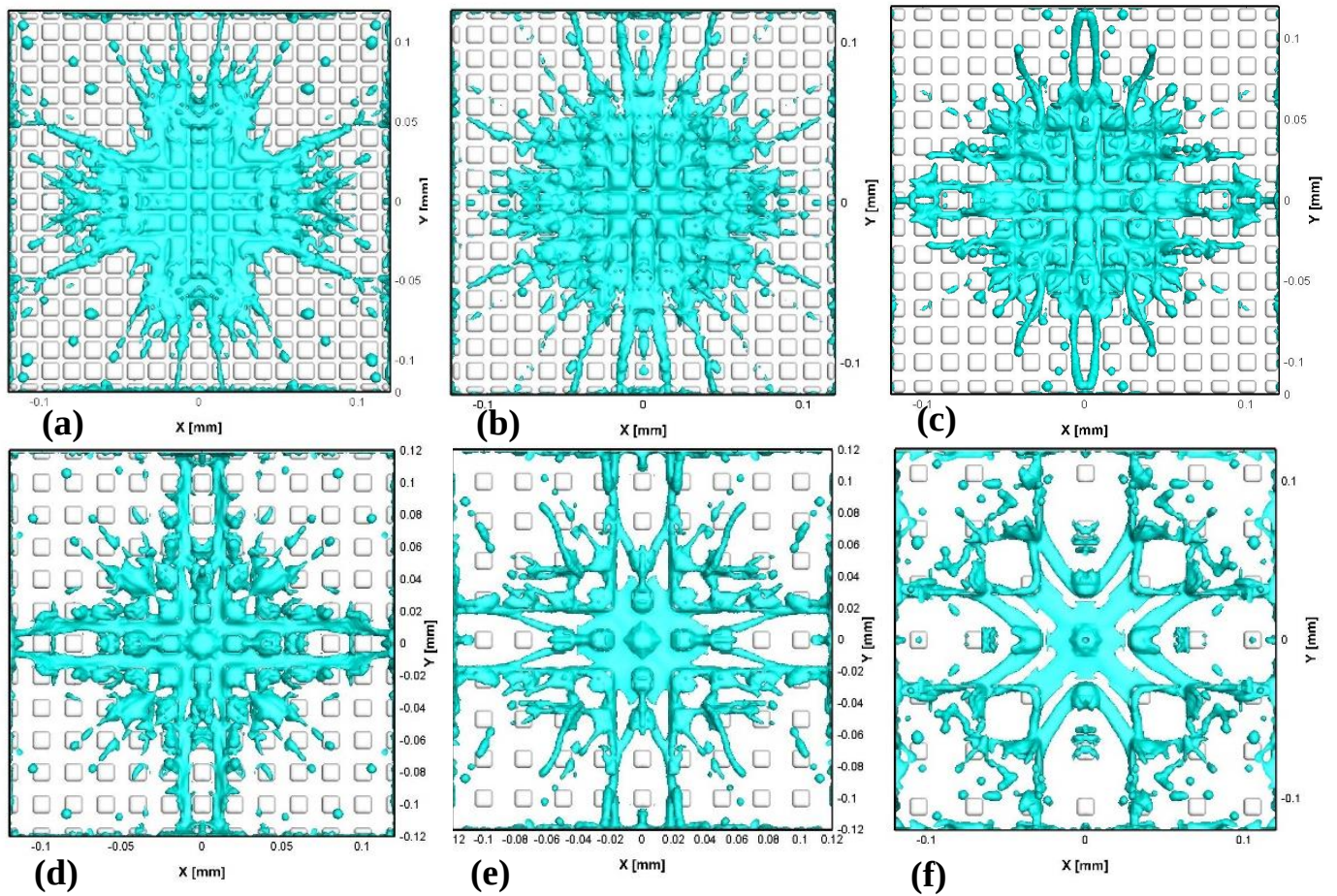


Figure 100. Simulated final splat morphology of Si impacting a pattern textured rb SiC substrate with increasing texture spacing: a) $3\mu\text{m}$, b) $5\mu\text{m}$, c) $7\mu\text{m}$, d) $10\mu\text{m}$, e) $15\mu\text{m}$, f) $25\mu\text{m}$. Other impact parameters are given in Table 23

observed that the smallest peak spacing in Figure 100a prevents adequate penetration of the droplet into the texture, causing splashing. When peak spacing is 10 μm or higher, Figure 100d-f, significant droplet flow between texture peaks is observed and splats become less coherent as the droplet is allowed to spread and splash.

Simulated splat spreading and solidification behavior on a textured surface is shown via 3D view in Figure 101. Figure 101a-f corresponds to the top views in Figure 100a-f 1 μs after initial impact. The blue regions of the impacting droplet represent solidified material, while the red regions are material which has not yet solidified. Increasing the texture spacing results in slowing the solidification of the impacting droplet in this case, and the droplet solidifies more rapidly when the texture spacing is small as the droplet is held in contact with the substrate by the texture resulting in greater heat transfer from droplet to substrate.

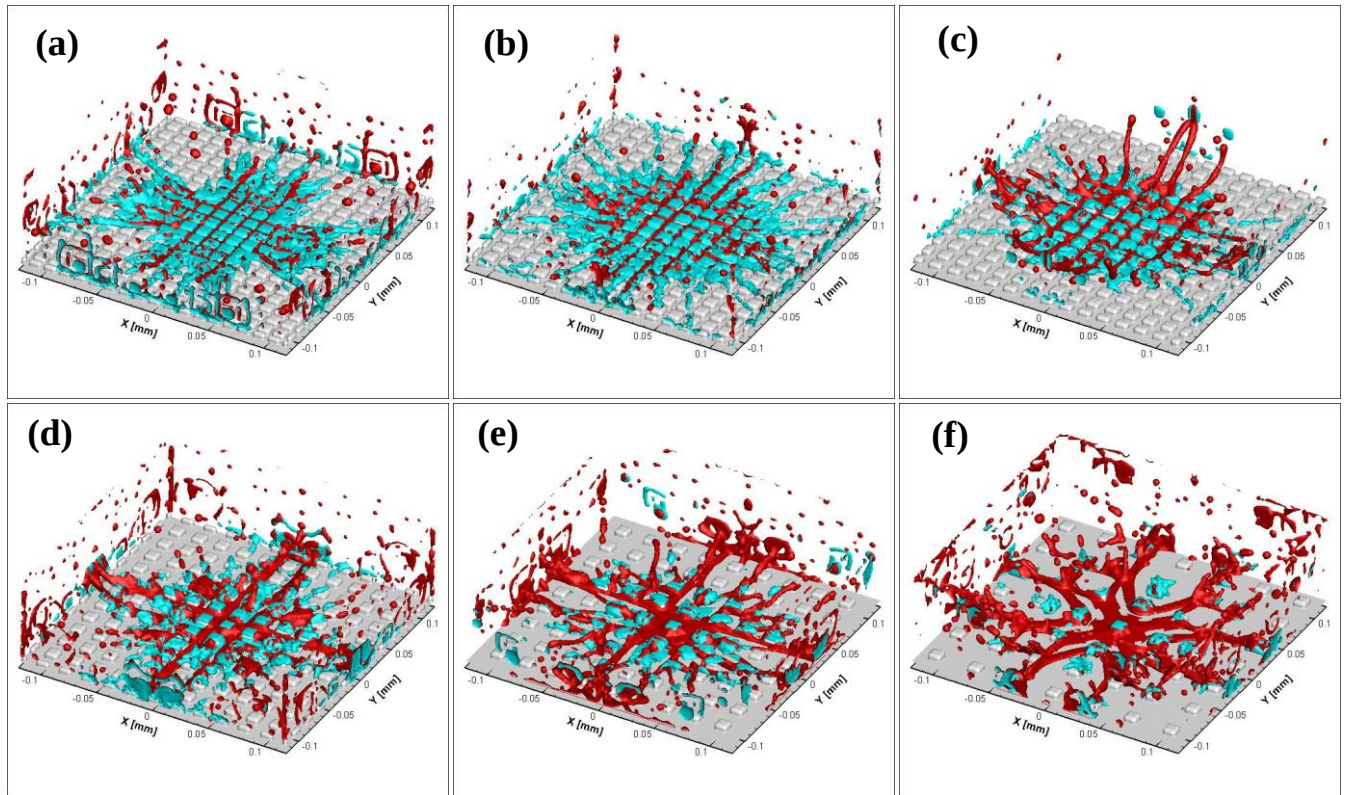


Figure 101. Simulated splat spreading, solidification and morphology of Si interaction with a pattern textured rb SiC substrate 1 μs after initial impact with increasing texture spacing: a) 3 μm , b) 5 μm , c) 7 μm , d) 10 μm , e) 15 μm , f) 25 μm . Blue material indicates solidified regions and red material has not solidified. Other impact parameters are given in Table 23

Figure 102 shows the effect of texture peak height on splat spreading and solidification via top and 3D view. Other impact and texture parameters are given in Table 23. The morphology of the

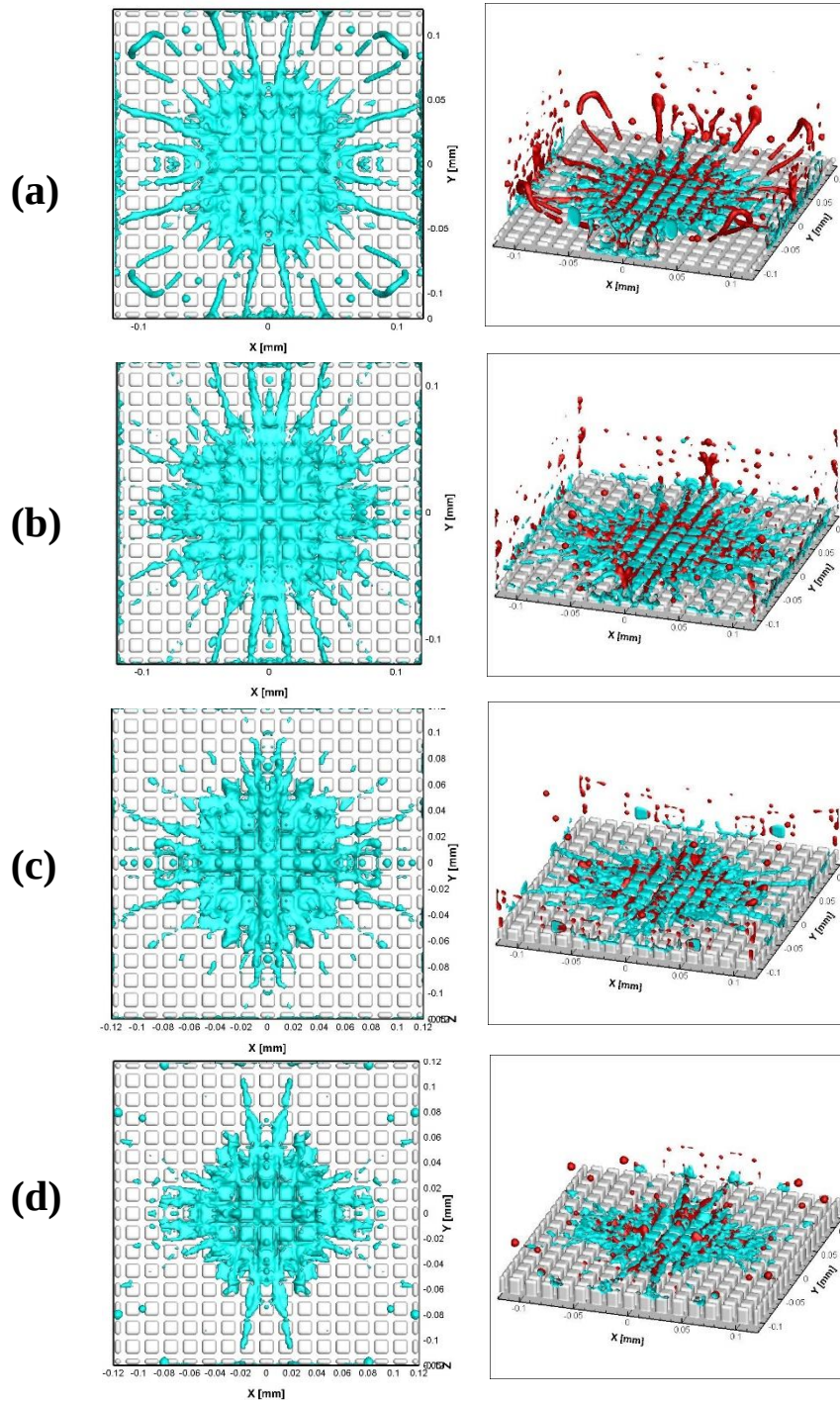


Figure 102. Top and 3D side view of simulated splat spreading, solidification and morphology of Si interaction with a pattern textured rb SiC substrate 1 μ s after initial impact with increasing texture peak height: a) 3 μ m, b) 5 μ m, c) 10 μ m, d) 15 μ m. . Blue material indicates solidified regions and red material has not solidified Other impact parameters are given in Table 23

splats remains similar with increasing peak height, although the amount of splashing and peripheral splat fingers decreases as the droplet flow is more significantly impeded by taller peaks. Though not as significant as increasing texture spacing, increasing peak height results in more rapid droplet solidification as the droplet is held in greater contact with the surface by the texture, further reducing splashing.

Figure 103 shows changes in splat morphology and solidification with increasing peak width from 5 to 10 to 15 μm with constant height and spacing. Overall splat morphology remains relatively consistent with changing peak width, though the smallest peak width in Figure 103a resulted in a diamond-shaped splat with shorter and fewer fingers at the periphery and the larger texture width in Figure 103b and c created rounder core splats with more and longer fingers. The droplet solidified more rapidly as peak width increased, as seen in Figure 103d-f, as the larger peak width provided a greater surface area for better contact between the droplet and substrate.

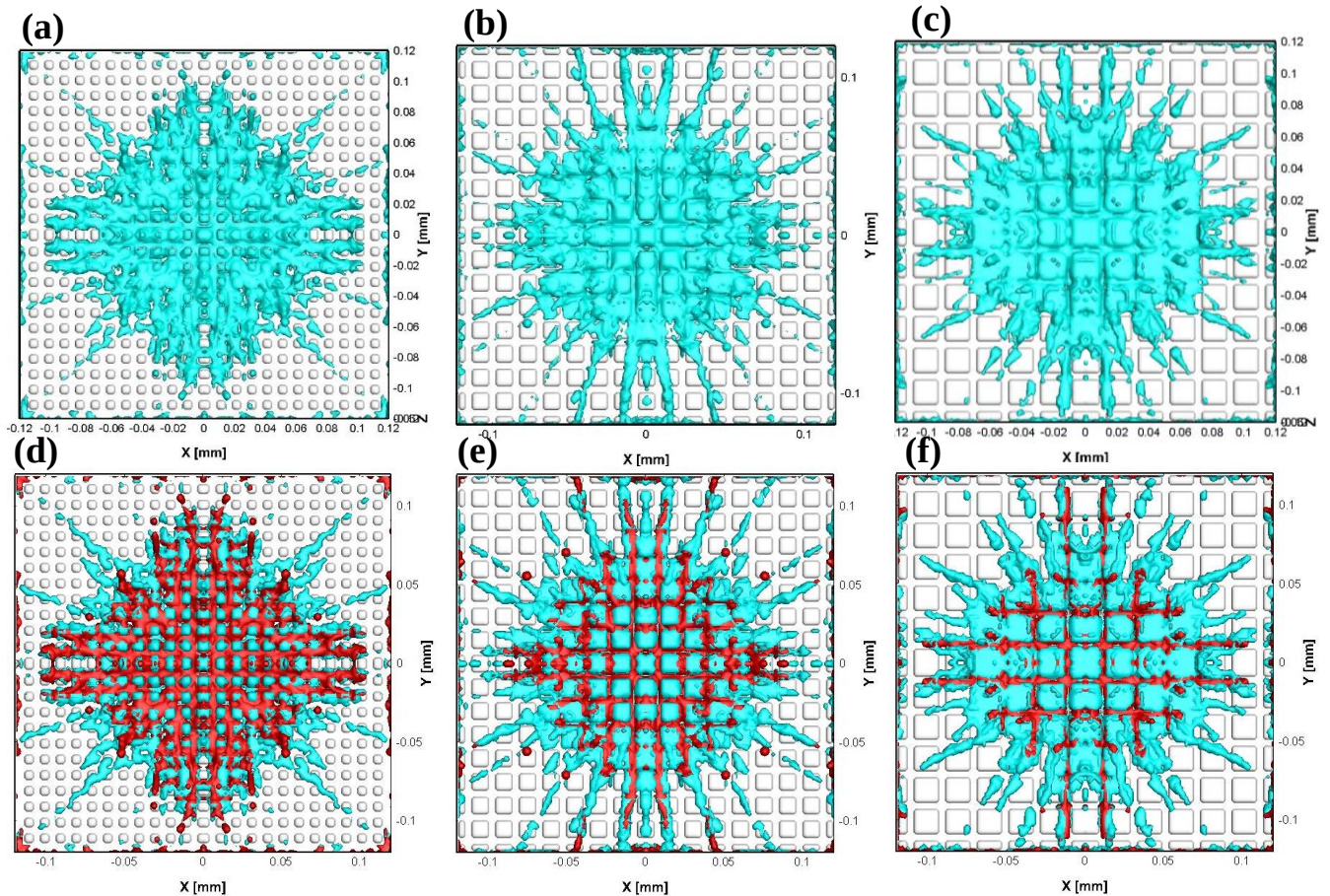


Figure 103. Simulated splat spreading, solidification and morphology of Si interaction with a pattern textured rb SiC substrate 1 μs after initial impact with increasing texture peak width: a&d) 5 μm , b&e) 10 μm , c&f) 15 μm . Blue material indicates solidified regions and red material has not solidified. Other impact parameters are given in Table 23

Based on the capabilities of the simulation software and a laser ablation technique at GF Machining Solutions, the surface texture patterns generated on actual rb SiC substrates via laser were recreated via simulation. Table 24 lists the texture parameters, peak height, width and spacing, for each pattern set and Figure 104 shows 3D topographical maps of laser textured surfaces and the corresponding simulated surface textures. For ease of discussion, texture patterns may be referred to based on peak dimensions as height-spacing-width. For example, 15-30-15 represents a texture pattern with peak height of 15 μm , spacing of 30 μm and width of 15 μm . Each peak parameter was varied at two levels, with the minimum peak spacing of 30 μm set by the capability of the laser texturing machine. As can be seen from the topographical maps in Figure 104, the peaks generated via laser have a Gaussian nature as the peak width increases from top to bottom, however measurements were made and verified that the peak dimensions were on target for each sample pattern.

Table 24. Texture pattern parameters for each sample set

Sample Pattern Set	Peak Height (μm)	Peak Spacing (μm)	Peak Width (μm)
1	15	30	15
2	15	30	30
3	30	30	15
4	30	30	30
5	15	45	15
6	15	45	30
7	30	45	15
8	30	45	30

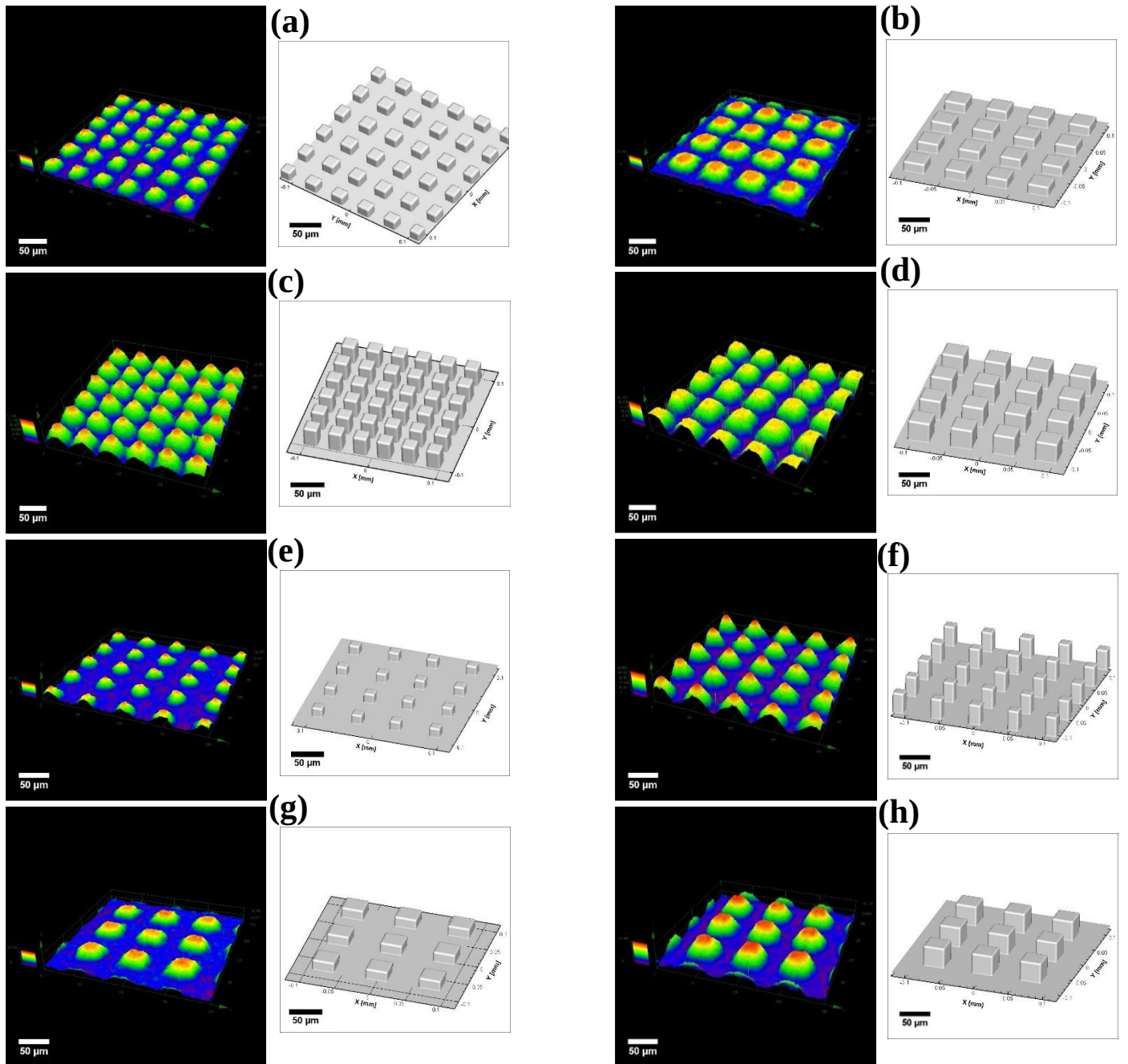


Figure 104. 3D topographical maps from a laser scanning microscope of laser textured rb SiC and corresponding simulated surface texture patterns with varying peak height, spacing and width (H-S-W in μm): a) 15-30-15, b) 15-30-30, c) 30-30-15, d) 30-30-30, e) 15-45-15, f) 30-45-15, g) 15-45-30, h) 30-45-30.

Table 25 provides the simulated droplet impact conditions based on measured parameters from recent plasma spraying of Si.

Table 25. Initial droplet conditions for simulating Si droplet impact on textured rb SiC during plasma spraying with varying texture parameters based on laser ablated substrate texture

Parameter	Value
Particle temperature (°C)	2340
Particle velocity (m/s)	354
Particle diameter (μm)	50
Substrate temperature, T _s , (°C)	140
Thermal contact resistance, TCR, (m ² K/W)	1x10 ⁻⁸
Texture peak height (μm)	15
Texture peak spacing (μm)	30
Texture peak width (μm)	15

The simulated evolution of splat spreading and solidification over time after impact on a textured rb SiC surface is shown in Figures 105 and 106. Figure 105b-d each provide a difference in level of peak width, height and spacing from the 15-30-15 pattern of Figure 105a, respectively, for comparison. Overall, the simulated spreading behavior is consistent despite variations in texture parameters as the impacting droplet spreads vertically and horizontally between the texture peaks. When the peak spacing is increased from 30 to 45 μm in Figure 105d, the nature of the droplet spreading changes slightly as the greater distance between peaks allows for more spreading in all directions, particularly after spreading surpasses the first set of peaks. More notably, the rate of solidification is seen to vary with texture parameters, especially peak spacing. Blue regions represent solidified material, while red regions represent not yet solidified material. The time at which solidification begins within the primary spreading droplet can be seen in Figures 105 and 106 when blue regions begin to form. Once again, the most substantial difference is seen when peak spacing is increased in Figure 105d, as well as Figure 106b-d. Solid regions in the primary splat in contact with the smooth, underlying surface begin forming after 0.5 μs and is mostly all solid after 0.75 μs, while solidification in the primary splat on a surface with peak spacing of 30 μm, Figure 105a-c, is first seen to begin after 0.75 μs. The increased spacing allows for greater droplet/surface contact in between the peaks and faster cooling. Also of note is when the peak height is increased to 30 μm in Figure 105c, the central region of the spreading droplet is more solidified after 0.75 μs than that of Figure 105a and b. The effect of each texture parameter on the mean splat cooling rate is plotted in Figure 107. The

mean cooling rate increases most significantly with the peak spacing, while changes in height and width have little effect.

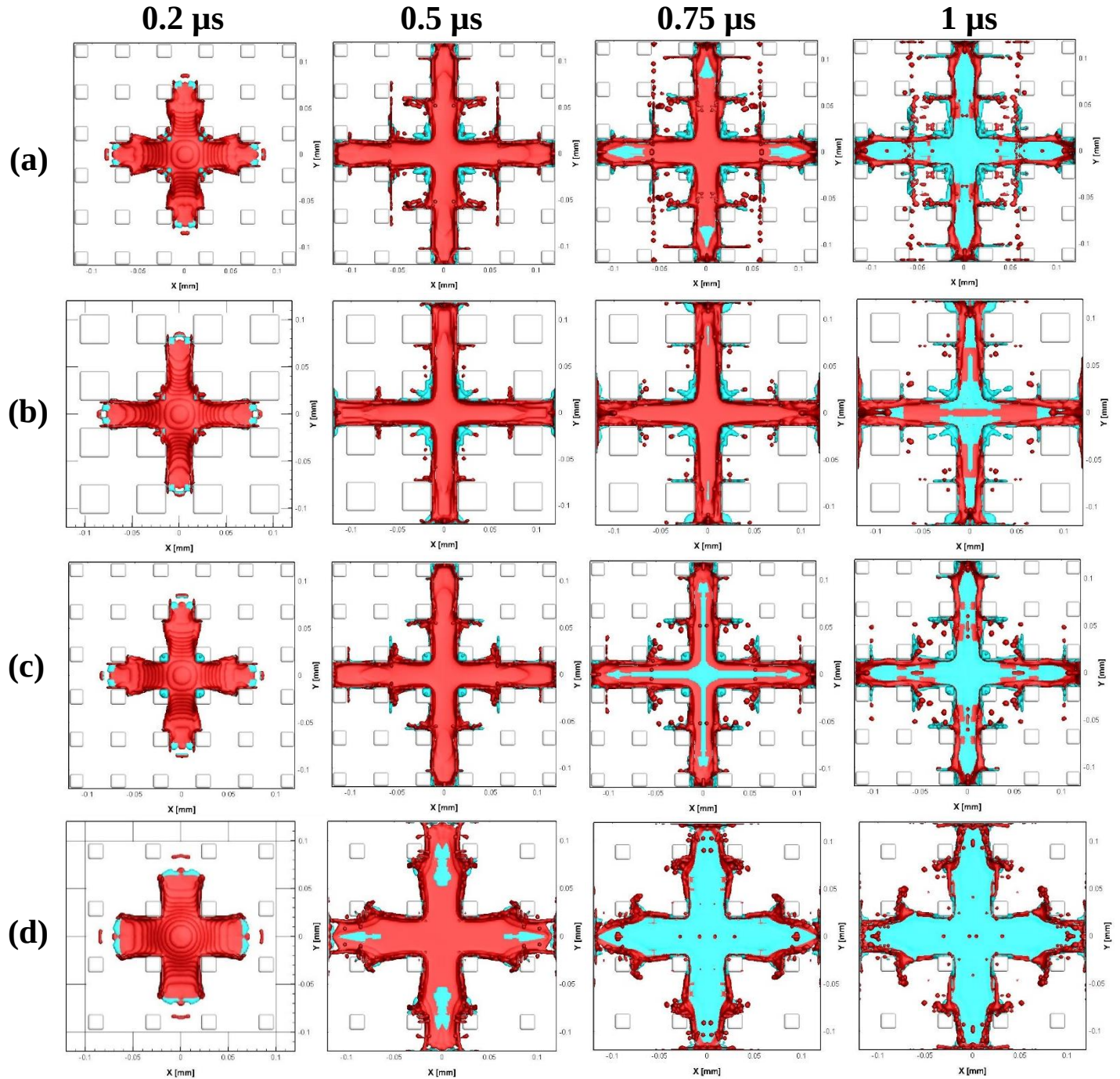


Figure 105. . Simulated splat spreading and solidification for Si interaction with a pattern textured rb SiC substrate at different times after initial impact with varying texture pattern: a) 15-30-15, b) 15-30-30, c) 30-30-15, d) 15-45-15. Other impact parameters are given in Table 25

In Figure 106, similar spreading and solidification patterns are observed with the increased texture spacing resulting in quicker solidification, and minor differences with varying peak height and width

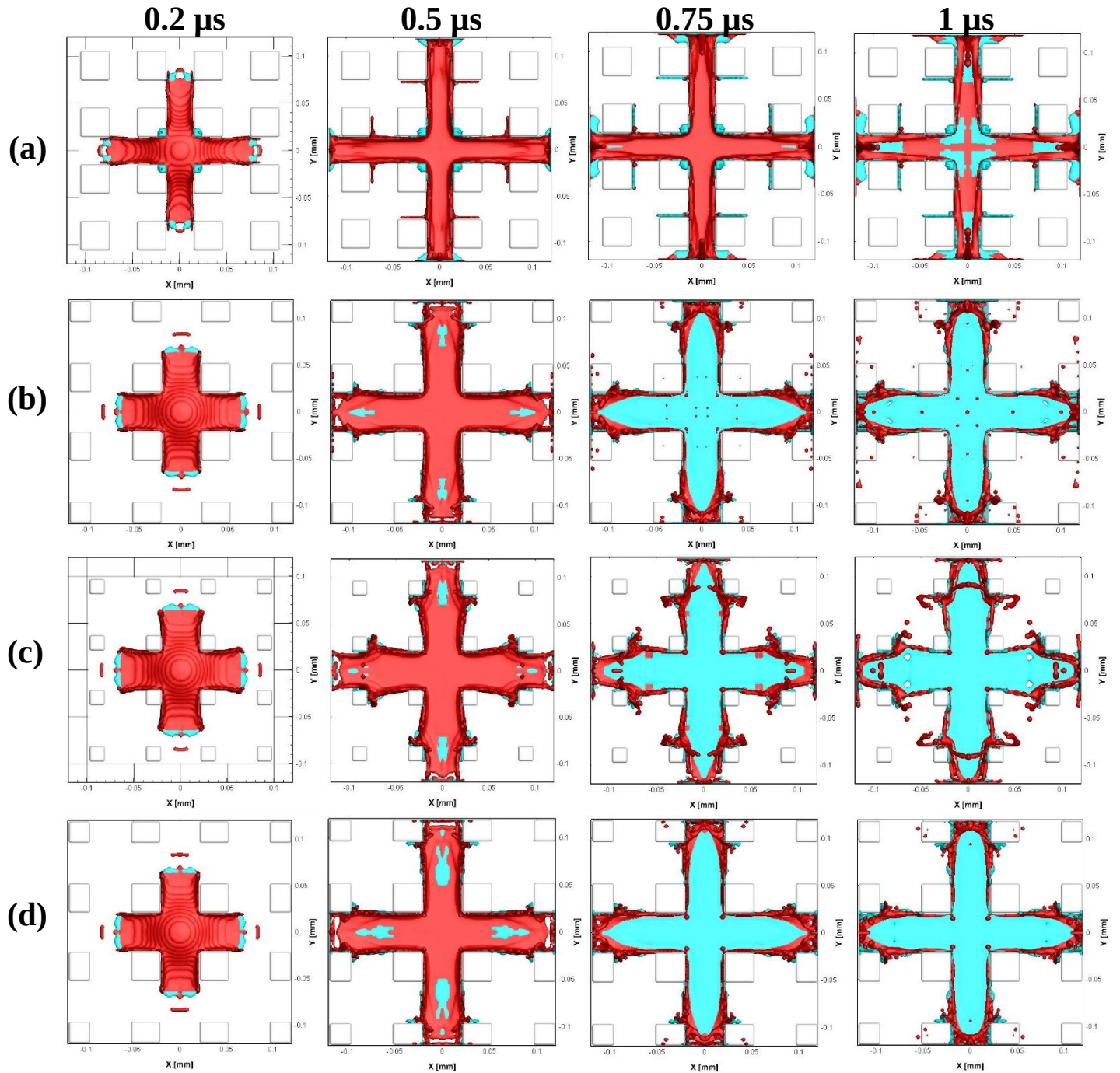


Figure 106. . Simulated splat spreading and solidification for Si interaction with a pattern textured rb SiC substrate at different times after initial impact with varying texture pattern: a) 30-30-30, b) 15-45-30, c) 30-45-15, d) 30-45-30. Other impact parameters are given in Table 25

The effect of texture height, spacing and width on the simulated mean splat cooling rate in the first 0.5 μs after impact is plotted in Figure 107. The cooling rate was found to be significantly affected by the texture spacing, while the height and width did not significantly affect the mean cooling rate. The splat cooling rate is important to consider as it relates to the spreading of the sprayed droplets within the texture, which could play a role in the ultimate adhesion of the coating to the substrate surface. The higher the cooling rate, the faster the droplet will solidify and the less it will spread within the texture. Whether this is a major factor in coating adhesion must be more deeply investigated, however should be noted. Figure 108 shows the simulated splat cooling rate for each texture pattern and the corresponding average coating adhesion strength. The smallest cooling rate is observed for pattern 4, 30-30-30, which also had the highest average adhesion strength. While splat cooling rate is not the only indicator of adhesion strength, in combination with other factors like surface roughness and spray parameters, it is a part of the full story

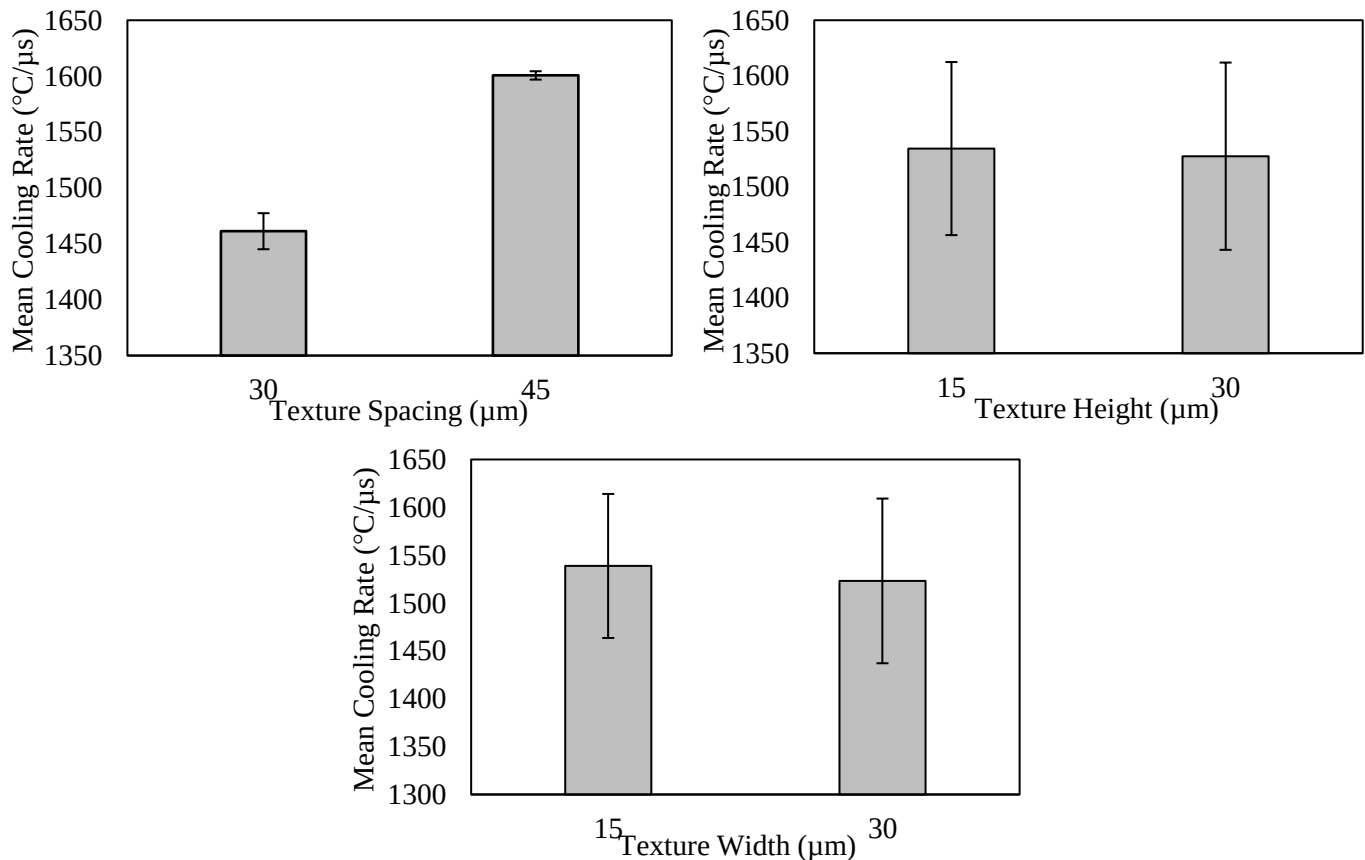


Figure 107. Mean simulated splat cooling rate as a function of varying texture spacing, height and width

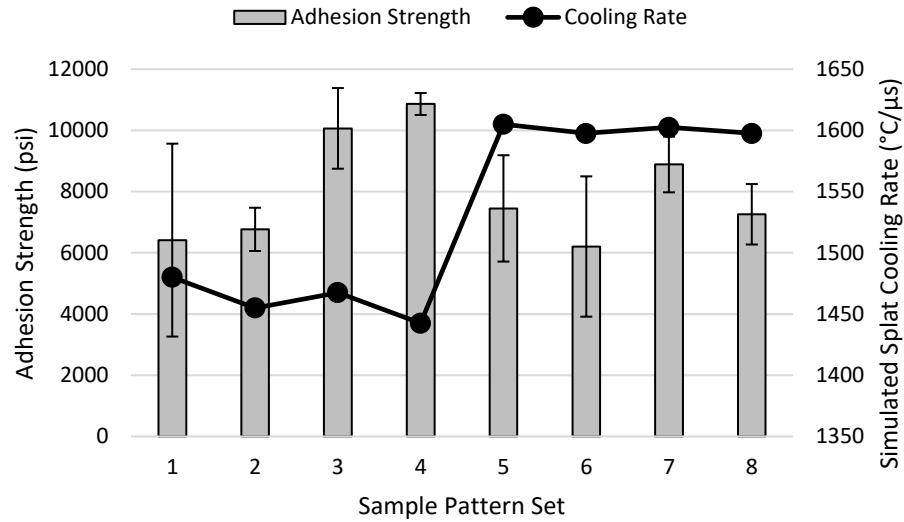


Figure 108. Plot of simulated splat cooling rate for each sample pattern set along with the corresponding measured coating adhesion strength

3.5.3 Summary

The experimental study of plasma spray coating behavior and splat formation is vital for making progress and optimizing process parameters to improve coating properties, however the time and financial cost associated with plasma spraying can be quite high. Therefore, simulating the plasma spray process and individual steps of the process like splat formation can be very helpful for increasing understanding of the effect of certain process variables and informing experimental studies. Simulation software SimDrop was utilized to model the impact, spreading and solidification of a plasma-sprayed Si droplet on a smooth SiC substrate as well as a substrate with varied regularly patterned texture parameters. Texture patterns comparable to texture generated on actual rb SiC substrates via laser ablation, which were subsequently plasma spray coated, were also simulated to observe Si splat impact and spreading behavior. Initial conditions for simulations were based on measured spray parameters for actual recent plasma spraying of Si. The effects of thermal contact resistance, substrate temperature, substrate thermal conductivity, droplet temperature and velocity on splat spreading and morphology were studied for Si on a smooth SiC surface. Experimental results from plasma spraying Si splats on smooth rb SiC substrates were compared to simulation results. The more disc-like splats observed on a

well preheated, smooth substrate corresponded to simulated results when experimental and simulation conditions closely matched and thermal contact resistance was reduced. On the other hand, the splat behavior, remnant shape and breakup observed experimentally on a non-preheated substrate matched simulation results when thermal contact resistance was increased, indicating the role substrate temperature plays in affecting droplet/substrate contact, thermal contact resistance and splat solidification.

Varying simulated substrate texture parameters peak height, spacing and width showed the effects of these parameters on splat spreading, shape and solidification. Each parameter was seen to affect splat formation and especially solidification. Peak spacing was seen to have the most dramatic effect on droplet spreading and solidification with larger spacing resulting in tremendous spreading, breakup, splashing and incoherence of splats, and smaller spacing limiting droplet spreading, speeding solidification and resulting in more compact splats. For simulated Si splat formation on surface texture patterns comparable to laser ablated surface texture, the peak spacing was seen to most significantly affect the cooling rate of splats relative to peak height and width. The surface texture which resulted in the slowest simulated splat cooling rate corresponded with the texture which had the highest experimentally measured Si coating adhesion strength, indicating the role that surface texture may play in splat solidification in addition to the well known role that texture and roughness play in mechanical interlocking of a plasma-sprayed coating and substrate.

4 Conclusions

The movement towards higher jet engine efficiency through increased operating temperature and reduced component weight has led to the utilization of SiC-based composites for some hot section components. These composites require environmental barrier coatings, likely with Si bond coatings, for use in the harsh engine environment. The relative novelty of these composites, their use for this application and the multi-layer environmental barrier coatings to be deposited on these materials leaves a gap in the knowledge and understanding of the effects of surface preparation and plasma spraying process parameters on the important, resulting coating properties.

This dissertation investigated the effects of automated grit blasting parameters on induced surface roughness and material/thickness loss of SiC composite substrates, as well as the adhesion strength of plasma-sprayed Si coatings on these substrates. Important grit blasting parameters for affecting roughness and thickness loss of these SiC composites were identified and utilizing high blast nozzle traverse speeds during automated grit blasting was found to significantly reduce thickness loss while maintaining a high degree of surface roughness. The role of substrate material and nature in grit blasting induced roughness and thickness loss was found to be substantial when blasting parameters were varied, and this must be taken into account during surface preparation.

The lack of studies of plasma spraying of Si on relevant substrates like SiC and SiC composites and especially on the very important property of Si coating adhesion strength and what affects it leaves a substantial knowledge gap in this field. This work also investigated the role of substrate preparation, surface roughness/texture and spray process parameters in plasma-sprayed Si coating adhesion strength and splat formation on rb SiC. A range of surface roughness was generated by varying grit blasting parameters prior to coating. The adhesion strength of the sprayed Si coatings was seen to be related to the induced roughness. Additionally, another method of generating a rough surface, laser ablation, was used to create controlled, regularly patterned surface texture on rb SiC substrates to further investigate the role of texture parameters in Si coating adhesion. The adhesion strength of the plasma sprayed coating was seen to be related to the varied texture parameters and the observations were discussed.

An established important factor in thermal spraying is the substrate temperature during coating deposition, as well as differences in substrate and coating materials. The role of rb SiC

substrate temperature in Si coating adhesion strength was evaluated for substrates roughened to the same degree. Coating adhesion strength increased dramatically with increasing substrate temperature when samples were preheated by the plasma torch prior to spraying. Substrate temperature was also varied for smooth rb SiC substrates on which single splats of Si were plasma sprayed to evaluate the effect on splat formation. Substrate temperature during spraying was seen to noticeably affect the number of splats adhering to the surface after spraying as well as the typical splat shape. Varying Si mean particle size affected in-flight particle properties during spraying and coating adhesion strength on grit blasted rb SiC. Results revealed that a Si mean particle size of 73 μm generated an average coating adhesion strength that was higher than that of a sprayed mean particle size of 56 or 94 μm , with the latter resulting in better adhesion strength than the former.

Simulations of Si droplet impact, spreading and solidification during plasma spraying on smooth and textured SiC surfaces were used to investigate the effects of relevant process parameters on splat formation. Experimentally observed Si splats on smooth substrates at different temperatures during deposition were matched with simulated splats with the same spraying parameters. A change in thermal contact resistance with changing substrate preheat temperature was confirmed by the simulation results. The role of surface texture parameters for a regularly patterned surface texture in splat formation was demonstrated through simulation. The pattern peak height, spacing and width all affected splat spreading, solidification and shape, with spacing and height having the greatest effect. Splat spreading and solidification on texture patterns modeled based on laser generated texture was simulated. The splat cooling rate was investigated with these simulations and was found to be affected by texture parameters, most significantly by the texture spacing. This highlights the importance of the role of surface roughness/texture on splat cooling and solidification as it relates to coating adhesion in addition to the mechanical interlocking of plasma sprayed coatings with substrate surfaces typically associated with surface roughness.

5 Future Work

In order to continue to narrow the knowledge gap that exists in understanding how to optimize surface preparation and plasma spray coating of SiC-based composites, much can be done to further investigate unanswered questions.

The significant role of substrate preheat temperature in plasma sprayed coating adhesion strength was made evident by this work and the work of others utilizing different substrate and coating materials. The existence of a transition temperature for the combination of a Si coating and SiC composite substrate at which coating adhesion dramatically increases is likely and should be further investigated by studying plasma sprayed coating adhesion strength and splat formation at a larger number of substrate preheat temperatures. On a similar note, something that does not seem to be mentioned or studied in general in this area is the combined effect of a range of substrate surface roughness and substrate temperature on coating adhesion strength. Observing whether coating adhesion strength can be significantly increased by controlling substrate temperature on smooth substrates would be very telling about the relative magnitude of the effects of substrate roughness and temperature on coating adhesion strength. Differentiating the effect of the immediate substrate temperature during coating deposition from the effects of substrate preheating on the surface, such as oxide layer growth or removal of contaminants, by preheating substrates to various temperatures and allowing them to cool prior to spraying and evaluating coating adhesion strength and/or splat formation would be telling about the mechanism of the preheating effect on Si coating adhesion strength and splat behavior. Similarly, it would be useful to perform this preheating via the same method for substrates which are not coated, and are stored in a way to protect the surface state until it can be characterized to understand what is changing about the surface. Little work has been done to investigate the effect of interaction between the plasma torch and substrate surfaces in general, let alone with SiC-based substrates. As the plasma torch preheating method is a quick and easy one, it is likely practiced often and knowing what effects it might be having that are different from more conventional heating methods, whether advantageous or not, would be beneficial. Comparing the results from more conventional preheating methods which are separate from the plasma torch prior to spraying would also be very valuable. Substrate temperature could be more easily

controlled and set with other heating methods compared to plasma torch preheating, which would allow for more controlled experiments to investigate temperature effects.

Advanced methods of monitoring and characterizing the coating and splat deposition process during plasma spraying would allow for greater understanding of the effects of process parameters on outcomes of interest like splat morphology and ultimately coating adhesion. The ability to measure the cooling rate of impacting particles on the surface via fast pyrometer and study which factors affect this cooling rate would provide much insight into the process, especially the role of substrate preheating and surface roughness in the initial interaction between plasma-sprayed particles and the substrate. This could also be very helpful for experimentally measuring thermal contact resistance between droplet and substrate and validating modeling observations.

Beyond the initial sprayed particles which form single splats, the subsequent sprayed particles and the buildup of the plasma-sprayed Si coating is also very important for the adhesion, cohesion and other resulting properties of the coating. The nature of the splat layering and coating formation should be studied experimentally and via simulation to gain a better understanding of how initial splats, substrate surface state and other process parameters may affect splat/splat interaction, interfaces and cohesion. Though coating/substrate adhesion is essential for a coating to perform as intended, its other properties are vital for its performance as well. Bulk, plasma-sprayed coating properties beyond adhesion/cohesion should be characterized simultaneously with the splat layering, interaction and coating buildup on a smaller scale to elucidate the effects of process factors.

References

- [1] P. Fauchais, J.V.R. Heberlein, and M. Boulos, *Thermal Spray Fundamentals: From Powder to Part*, Springer, New York, 2014
- [2] B. J. Griffiths, D. T. Gawne and G. Dong, A Definition of the Topography of Grit-Blasted Surfaces for Plasma Sprayed Alumina Coatings, *Journal of Manufacturing Science and Engineering*, February 1999, **121**, p 49-53
- [3] M. Mellali, P. Fauchais, A. Grimaud, Influence of substrate roughness and temperature on the adhesion/cohesion of alumina coatings, *Surface and Coatings Technology*, 1996, **81**, p 275-286
- [4] D. Sen, N.M. Chavan, D.S. Rao and G. Sundararajan, Influence of Grit Blasting on the Roughness and the Bond Strength of Detonation Sprayed Coating, *Journal of Thermal Spray Technology*, June 2010, **19**(4), p 805-815
- [5] S. K. Asl and M. H. Sohi, Effect of grit-blasting parameters on the surface roughness and adhesion strength of sprayed coating, *Surf. Interface Anal*, 2010, **42**, p 551-554
- [6] K. Bobzin, M. Ote, T.F. Linke, J. Sommer and X. Liao, Influence of Process Parameter on Grit Blasting as a Pretreatment Process for Thermal Spraying, *Journal of Thermal Spray Technology*, January 2016, **25**(1-2), p 3-11
- [7] K. Bobzin, M. Ote, M. A. Knoch, Surface Pre-treatment for Thermally Sprayed ZnAl15 Coatings, *Journal of Thermal Spray Technology*, 2017, **26**, p 464-472
- [8] D. Zhu, Aerospace Ceramic Materials: Thermal, Environmental Barrier Coatings and SiC/SiC Ceramic Matrix Composites for Turbine Engine Applications, *National Aeronautics and Space Administration*, Glenn Research Center, Cleveland, OH, May 2018
- [9] E.J. Opila and R. Hann, "Paralinear Oxidation of CVD SiC in Water Vapor," *Journal of American Ceramic Society*, vol. 80, no. 1, pp. 197-205, 1997
- [10] A. Nistal, E. García, C. García-Diego, M. I. Osendi, and P. Miranzo, "Flame spraying of adherent silicon coatings on SiC substrates," *Surface and Coatings Technology*, vol. 270, pp. 8–15, 2015, doi: 10.1016/j.surfcoat.2015.03.025.
- [11] K.N. Lee, *Surf. Coat. Technol.* 133–134 (2000) 1–7.
- [12] K.N. Lee, D.S. Fox, J.I. Eldridge, D. Zhu, R.C. Robinson, N.P. Bansal, R.A. Miller, *J. Am. Ceram. Soc.* 86 (2003) 1299–1306.
- [13] N.S. Jacobson, *J. Am. Ceram. Soc.* 76 (1993) 3–28.
- [14] K.N. Lee, R.A. Miller, *Surf. Coat. Technol.* 86–87 (1996) 142–148.
- [15] Y.S. Touloukian, R.K. Kirby, R.E. Taylor, T.Y.R. Lee, *Thermophysical Properties of Matter*, vol. 13, IFI/Plenum, New York, 1977.
- [16] Y. Niu, X. Liu, X. Zheng, H. Ji, and C. Ding, "Microstructure and properties characterization of silicon coatings prepared by vacuum plasma spraying technology," *Journal of Thermal Spray Technology*, vol. 18, no. 3, pp. 427–434, 2009, doi: 10.1007/s11666-009-9326-1.
- [17] Y. Niu, X. Zheng, X. Liu, H. Ji, and C. Ding, "Influence of powder size on characteristics of air plasma sprayed silicon coatings," *Ceramics International*, vol. 38, no. 7, pp. 5897–5905, 2012, doi: 10.1016/j.ceramint.2012.03.080.

- [18] Y. Niu, X. Liu, and C. Ding, "Phase composition and microstructure of silicon coatings deposited by air plasma spraying," *Surface and Coatings Technology*, vol. 201, no. 3–4, pp. 1660–1665, 2006, doi: 10.1016/j.surfcoat.2006.02.040.
- [19] E. García *et al.*, "Thermally Sprayed Y₂O₃-Al₂O₃-SiO₂ Coatings for High-Temperature Protection of SiC Ceramics," *Journal of Thermal Spray Technology*, vol. 24, no. 1–2, pp. 185–193, 2014, doi: 10.1007/s11666-014-0178-y.
- [20] B. (Dave) Kharas, G. Wei, S. Sampath, and H. Zhang, "Morphology and microstructure of thermal plasma sprayed silicon splats and coatings," *Surface and Coatings Technology*, vol. 201, no. 3–4, pp. 1454–1463, 2006, doi: 10.1016/j.surfcoat.2006.02.014.
- [21] H.R. Khan, H. Frey, H. Schoellkopf, "R.f. plasma spray and ion beam deposition of microcrystalline and amorphous silicon films and their morphological, structural and photoconducting properties," *Surface and Coatings Technology*, vol. 67, no. 1-2, (1994) 79-83
- [22] R. Suryanarayanan, G. Brun, M. Akani, *Thin Solid Films* 119 (1984) 67–73.
- [23] M. Akani, R. Suryanarayanan, G. Brun, Resistivity and photoconductivity of plasma sprayed polycrystalline silicon, 151 (1987) 343–353.
- [24] F. Tamura, Y. Okayasu, K. Kumagai, *Sol. Energy Mater. Sol. Cells* 34 (1994) 263–270.
- [25] M. Akani, R. Suryanarayanan, and G. Brun, Influence of Process Parameters on the Electrical Properties of Plasma-Sprayed Silicon, *J. Appl. Phys.*, 1986, 60(1), p 457-459
- [26] R. Suryanarayanan, M. Akani, R. Gauthier, R.M. Ghaieth, and P. Pinard, Electron Beam Recrystallization of Plasma-Sprayed Silicon Substrates, *Appl. Phys. Lett.*, 1987, 51, p 259-260
- [27] S. Sivoththaman, M. Rodot, R. Suryanarayanan, and A. Eyer, Optical Lamp Recrystallization of Plasma Sprayed Silicon Deposits on Different Substrates, *Mater. Res. Bull.*, 1992, 27, p 425-430
- [28] B.D. Kharas, S. Sampath, and R.J. Gambino, Anisotropic Resistivity in Plasma-Sprayed Silicon Thick Films, *J. Appl. Phys.*, 2005, 97, p 094906-1
- [29] S. Sampath, A. Patel, A.H. Dent, R. Gambino, H. Herman, R. Greenlaw, and E. Tormey, Materials for Direct Write Electronics, *MRS Bull.*, 2000, 25, p 181-184
- [30] R.B. Bergmann and J.H. Werner, The Future of Crystalline Silicon Films on Foreign Substrates, *Thin Solid Films*, 2002, 403-404, p 162-169
- [31] L. Bianchi, A. C. Leger, M. Vardelle, A. Vardelle, and P. Fauchais, "Splat formation and cooling of plasma-sprayed zirconia," *Thin Solid Films*, vol. 305, no. 1–2, pp. 35–47, 1997, doi: 10.1016/S0040-6090(97)80005-3.
- [32] X. Jiang, J. Matejcek, and S. Sampath, "Substrate temperature effects on the splat formation, microstructure development and properties of plasma sprayed coatings: Part II: Case study for molybdenum," *Materials Science and Engineering A*, vol. 272, no. 1, pp. 189–198, 1999, doi: 10.1016/S0921-5093(99)00461-X.
- [33] S. Sampath and X. Jiang, "Splat formation and microstructure development during plasma spraying: Deposition temperature effects," *Materials Science and Engineering A*, vol. 304–306, no. 1–2, pp. 144–150, 2001, doi: 10.1016/S0921-5093(00)01464-7.
- [34] M. Fukumoto, T. Yamaguchi, M. Yamada, and T. Yasui, "Splash splat to disk splat transition behavior in plasma-sprayed metallic materials," *Journal of Thermal Spray Technology*, vol. 16, no. 5–6, pp. 905–912, 2007, doi: 10.1007/s11666-007-9083-y.

- [35] M. Fukumoto, H. Nagai, and T. Yasui, "Influence of surface character change of substrate due to heating on flattening behavior of thermal sprayed particles," *Proceedings of the International Thermal Spray Conference*, vol. 15, no. December, pp. 759–764, 2006, doi: 10.1361/105996306X146776.
- [36] M. Fukumoto, K. Yang, K. Tanaka, T. Usami, T. Yasui, and M. Yamada, "Effect of substrate temperature and ambient pressure on heat transfer at interface between molten droplet and substrate surface," *Journal of Thermal Spray Technology*, vol. 20, no. 1–2, pp. 48–58, 2011, doi: 10.1007/s11666-010-9537-5.
- [37] M. FUKUMOTO, K. YANG, M. YAMADA, and T. YASUI, "Splat Formation Mechanism in Thermal Spraying," *Journal of Solid Mechanics and Materials Engineering*, vol. 5, no. 12, pp. 1001–1014, 2011, doi: 10.1299/jmmp.5.1001.
- [38] K. Yang, M. Liu, and C. Deng, "Adhesion strength of Cu coating on substrate at different temperatures," *Surface Engineering*, vol. 30, no. 11, pp. 814–821, 2014, doi: 10.1179/1743294414Y.0000000320.
- [39] Y. Zhang, S. Matthews, and M. Hyland, "Understanding the formation of plasma-sprayed Ni20Cr splats through interface observation," *Applied Surface Science*, vol. 469, no. November 2018, pp. 691–702, 2019, doi: 10.1016/j.apsusc.2018.11.087.
- [40] Q. Wei, L. He, Z. Liu, X. Feng, and Y. Z. Xing, "The Morphology Analysis of Plasma-Sprayed Cast Iron Splats at Different Substrate Temperatures via Fractal Dimension and Circularity Methods," *High Temperature Materials and Processes*, vol. 38, no. 2019, pp. 692–698, 2019, doi: 10.1515/htmp-2019-0021.
- [41] D. K. Christoulis, D. I. Pantelis, N. de Dave-Fabrègue, F. Borit, V. Guipont, and M. Jeandin, "Effect of substrate temperature and roughness on the solidification of copper plasma sprayed droplets," *Materials Science and Engineering A*, vol. 485, no. 1–2, pp. 119–129, 2008, doi: 10.1016/j.msea.2007.07.059.
- [42] A. T. T. Tran, M. M. Hyland, T. Qiu, B. Withy, and B. J. James, "Effects of surface chemistry on splat formation during plasma spraying," *Journal of Thermal Spray Technology*, vol. 17, no. 5–6, pp. 637–645, 2008, doi: 10.1007/s11666-008-9237-6.
- [43] A. T. T. Tran, M. M. Hyland, K. Shinoda, and S. Sampath, "Influence of substrate surface conditions on the deposition and spreading of molten droplets," *Thin Solid Films*, vol. 519, no. 8, pp. 2445–2456, 2011, doi: 10.1016/j.tsf.2010.11.047.
- [44] V. Pershin, M. Lufitha, S. Chandra, and J. Mostaghimi, "Effect of substrate temperature on adhesion strength of plasma-sprayed nickel coatings," *Journal of Thermal Spray Technology*, vol. 12, no. 3, pp. 370–376, 2003, doi: 10.1361/105996303770348249.
- [45] Haure T. Multifunctional layers obtained by a multi-technique process, Ph.D. University of Limoges, France, Nov 2003
- [46] Syed AA, Denoirjean A, Hannoyer B, Fauchais P, Denoirjean P, Khan AA, Labbe JC (2005) Influence of substrate surface conditions on the plasma sprayed ceramic and metallic particles flattening. *Surf Coat Technol* 200:2317–2331
- [47] Pech J (1999) Preoxidation generated by blown arc d.c. plasma jets. Relationship between surface, oxidation and adhesion of plasma sprayed coatings. Ph.D., University of Rouen, France
- [48] Pech J, Hannoyer B (2000) Influence of the oxide layer by d.c. plasma preheating on the adhesion coating and role of the initial surface pretreatment. *Surf Interface Anal* 30:585–588

- [49] M. F. Bahbou, P. Nylén, Relationship between surface topography parameters and adhesion strength for plasma spraying, *International Thermal Spray Conference & Exposition*, May 2005, Conference proceedings, p 1-5
- [50] B. J. Griffiths, D. T. Gawne and G. Dong, The role of grit blasting in the production of high-adhesion plasma sprayed alumina coatings, *Proc Instn Mech Engrs*, 1997, **211**(1), p 1-9
- [51] M. F. Bahbou, P. Nylén and J. Wigren, Effect of Grit Blasting and Spraying Angle on the Adhesion Strength of a Plasma-Sprayed Coating, *Journal of Thermal Spray Technology*, December 2004, **13**(4), p 508-514
- [52] R. Kromer, J. Cormier, S. Costil, Role of Powder Granulometry and Substrate Topography in Adhesion Strength of Thermal Spray Coatings, *Journal of Thermal Spray Technology*, Vol. 25(5), (2016), 933-945
- [53] N. Tan, Z. Xing, X. Wang, H. Wang, G. Jin, S. Chen, B. Xu, Deposition mechanism of plasma sprayed droplets on textured surfaces with different diameter-to distance ratios, *Materials and Design*, 2017, **133**, p 19-29
- [54] N. Tan, Z. Xing, X. Wang, H. Wang, G. Jin, B. Xu, Effects of texturing patterns on the adhesion strength of atmosphere plasma sprayed coatings, *J. Mater. Res.*, May 2017, **32**(9), p 1682-1692
- [55] N. Tan, Z. Xing, X. Wang, H. Wang, G. Jin, B. Xu, Investigation of sprayed particle filling qualities within the texture on the bonding behavior of Ni-based coating, *Surface & Coatings Technology*, 2017, **330**, p 131-139
- [56] R. Kromer, J. Cormier, S. Costil, Laser surface patterning to enhance adhesion of plasma sprayed coatings, *Surface & Coatings Technology*, 2015, **278**, p 171-182
- [57] S. Costil, S. Lukat, P. Bertrand, C. Langlade, and C. Coddet, "Surface treatment effects on ceramic matrix composites: Case of a thermal sprayed alumina coating on SiC composites," *Surface and Coatings Technology*, vol. 205, no. 4, pp. 1047–1054, 2010, doi: 10.1016/j.surfcoat.2010.07.021
- [58] H. Begg, M. Riley, H. V. Lovelock, Mechanization of the Grit Blasting Process for Thermal Spray Coating Applications: A Parameter Study, *Journal of Thermal Spray Technology*, January 2016, **25**(1-2), p 12-20
- [59] B. J. Griffiths, D. T. Gawne, G. Dong, The erosion of steel surfaces by grit-blasting as a preparation for plasma spraying, *Wear*, 1996, **194**, p 95-102
- [60] J. Day, X. Huang, N. L. Richards, Examination of a Grit-Blasting Process for Thermal Spraying Using Statistical Methods, *Journal of Thermal Spray Technology*, December 2005, **14**(4), p 471-479
- [61] M. Mellali, A. Grimaud, A.C. Leger, P. Fauchais, J. Lu, Alumina Grit Blasting Parameters for Surface Preparation in the Plasma Spraying Operation, *Journal of Thermal Spray Technology*, Vol. 6(2), (1997), 217-227.
- [62] J. C. Miller, *Laser Ablation: Principles and Applications*, Springer Series in Materials Science, **28**, Berlin, 1994
- [63] E.S. Gadelmawla, M.M. Koura, T.M.A. Maksoud, I.M. Elewa, H.H. Soliman, Roughness parameters, *Journal of Materials Processing Technology*, Volume 123, Issue 1, 2002, Pages 133-145,
- [64] Trelleborg Sealing Solutions, Surface Measurement Methods, Aerospace Engineering Guide, April 2004, p 21

- [65] ISO 4287:1997, Geometrical Product Specifications (GPS) -- Surface texture: Profile method -- Terms, definitions and surface texture parameters
- [66] G. Montavon, Quality control of thermal sprayed coatings, Course Thermal Spraying, (2004), Aliderte Limoges France
- [67] A.C. Leger, M. Vardelle, A. Vardelle, P. Fauchais, S. Sampath, C.C. Berndt, H. Herman, in: C. Berndt (Ed.), Practical Solution for Engineering Problem, ASM International, Metals Park, OH, 1996, p. 623.
- [68] H. Li, S. Costil, H. L. Liao, C. J. Li, M. Planche, and C. Coddet, "Effects of surface conditions on the flattening behavior of plasma sprayed Cu splats," *Surface and Coatings Technology*, vol. 200, no. 18–19, pp. 5435–5446, 2006, doi: 10.1016/j.surfcoat.2005.07.058.
- [69] C.-J. Li, H.-L. Liao, P. Gougeon, G. Montavon, C. Coddet, in: C. Moreau, B. Marple (Eds.), Thermal Spray 2003: Advancing the Science and Applying the Technology, ASM International, Materials Park, Ohio, USA, 2003, p. 875.
- [70] Y. Tanaka and M. Fukumoto, "Investigation of dominating factors on flattening behavior of plasma sprayed ceramic particles," *Surface and Coatings Technology*, vol. 120–121, pp. 124–130, 1999, doi: 10.1016/S0257-8972(99)00348-5.
- [71] M. Fukumoto, Y. Huang, *J. Therm. Spray Technol.* 8 (1999) 427.
- [72] Z. Feng, M. Domaszewski, G. Montavon, C. Coddet, Finite element analysis of effect of substrate surface roughness on liquid droplet impact and flattening process, *Journal of Thermal Spray Technology*, Vol. 11(1), (2002), 62-68.
- [73] C. Moreau, P. Gougeon, and M. Lamontagne, "Influence of substrate preparation on the flattening and cooling of plasma-sprayed particles," *Journal of Thermal Spray Technology*, vol. 4, no. 1, pp. 25–33, 1995, doi: 10.1007/BF02648525.
- [74] K. Shinoda, A. Yamada, M. Kambara, Y. Kojima, and T. Yoshida, "Deformation of alumina droplets on micro-patterned substrates under plasma spraying conditions," *Journal of Thermal Spray Technology*, vol. 16, no. 2, pp. 300–305, 2007, doi: 10.1007/s11666-007-9033-8.
- [75] V. Pershin, I. Thompson, S. Chandra, and J. Mostaghimi: "Splashing of Nickel Droplets During Plasma Spraying" in *Thermal Spray Surface Engineering via Applied Research*, C. Berndt, ed., ASM International, Materials Park, OH, 2000, pp. 721-23.
- [76] S. Sampath, H. Herman, *J. Thermal Spray Technol.* 4 (1996) 445.
- [77] R. Ghafouri-Azar, S. Chandra, J. Mostaghimi. ILASS Americas, 14th Annual Conference on Liquid Atomization and Spray Systems, Dearborn, MI, May 2001.
- [78] M. Bussmann, S. Chandra, J. Mostaghimi. *Phys. Fluids* 12, 3121–3132 (2000).
- [79] M. Bussmann, J. Mostaghimi, S. Chandra. *Phys. Fluids* 11, 1406–1417 (1999).
- [80] M. Pasandideh-Fard, V. Pershin, S. Chandra, J. Mostaghimi. *J. Thermal Spray Technol.* (2002). In press.
- [81] J. Mostaghimi, M. Pasandideh-Fard, S. Chandra. *Plasma Chem. Plasma. Processing* 22, 59–84 (2001).
- [82] M. Pasandideh-Fard, S. Chandra, and J. Mostaghimi, "A three-dimensional model of droplet impact and solidification," *International Journal of Heat and Mass Transfer*, vol. 45, no. 11, pp. 2229–2242, 2002, doi: 10.1016/S0017-9310(01)00336-2.

- [83] J. Mostaghimi and S. Chandra, "Splat formation in plasma-spray coating process," *Pure and Applied Chemistry*, vol. 74, no. 3, pp. 441–445, 2002, doi: 10.1351/pac200274030441.
- [84] K. Shinoda, Y. Kojima, and T. Yoshida, In Situ Measurement System for Deformation and Solidification Phenomena of Yttria- Stabilized Zirconia Droplets Impinging on Quartz Glass Substrate Under Plasma-Spraying Conditions, *J. Thermal Spray Technol.*, 2005, 14(4), p 511-517
- [85] H. B. Parizi, L. Rosenzweig, J. Mostaghimi, S. Chandra, T. Coyle, H. Salimi, L. Pershin, A. McDonald, C. Moreau, Numerical simulation of droplet impact on patterned surfaces, *Journal of Thermal Spray Technology*, 2008, 16(5–6), p 713–721
- [86] K. Shinoda, M. Raessi, J. Mostaghimi, T. Yoshida, and H. Murakami, "Effect of substrate concave pattern on splat formation of yttria-stabilized zirconia in atmospheric plasma spraying," *Journal of Thermal Spray Technology*, vol. 18, no. 4, pp. 609–618, 2009, doi: 10.1007/s11666-009-9374-6.
- [87] M. Raessi, J. Mostaghimi, and M. Bussmann, "Effect of surface roughness on splat shapes in the plasma spray coating process," *Thin Solid Films*, vol. 506–507, pp. 133–135, 2006, doi: 10.1016/j.tsf.2005.08.140.
- [88] SimDrop 4.1 Manual, Droplet Impact and Solidification Software, Simulent, Inc., Toronto, Ont.
- [89] ISO, 25178-2: 2012. Geometrical product specifications (GPS)— Surface texture: Areal—Part 2: Terms, definitions and surface texture parameters.
- [90] R. Deltombe, K. J. Kubiak, and M. Bigerelle, "How to select the most relevant 3D roughness parameters of a surface," *Scanning*, vol. 36, no. 1, pp. 150–160, 2014, doi: 10.1002/sca.21113.
- [91] "ASTM Designation C 633-Standard Method of Test for Adhesion or Cohesive Strength of Flame-Sprayed Coatings" in *Annual Book of ASTM Standards*, Section 15, Vol. 15.02, American Society for Testing and Materials, Philadelphia, PA, 1985, pp. 340-46.
- [92] R. Kromer, S. Costil, C. Verdy, S. Gojon, and H. Liao, "Laser surface texturing to enhance adhesion bond strength of spray coatings – Cold spraying, wire-arc spraying, and atmospheric plasma spraying," *Surface and Coatings Technology*, vol. 352, pp. 642–653, 2018, doi: 10.1016/j.surfcoat.2017.05.007.
- [93] R. Kromer, S. Costil, J. Cormier, L. Berthe, P. Peyre, and D. Courapied, Laser Patterning Pretreatment Before Thermal Spraying: A Technique to Adapt and Control the Surface Topography to Thermomechanical Loading and Materials, *J. Therm. Spray Technol.*, 2016, 25(3), p 401-410
- [94] Y. Zhang, C. Y. Hsu, S. Aubuchon, P. Karandikar, and C. Ni, "Microstructural and thermal property evolution of reaction bonded silicon carbide (RBSC)," *Journal of Alloys and Compounds*, vol. 764, pp. 107–111, 2018, doi: 10.1016/j.jallcom.2018.05.321.
- [95] Y. B. Magomedov and G. G. Gadjeiev, "High-temperature thermal conductivity of silicon in the solid and liquid states," *High Temperature*, vol. 46, no. 3, pp. 422–424, 2008, doi: 10.1134/S0018151X08030206.
- [96] S. I. Chung, K. Izunome, A. Yokotani, and S. Kimura, "Estimation of surface tension of molten silicon using a dynamic hanging drop," *Japanese Journal of Applied Physics*, vol. 34, no. 5, pp. L631–L634, 1995, doi: 10.1143/JJAP.34.L631.
- [97] H. Sasaki, E. Tokizaki, X. M. Huang, K. Terashima, and S. Kimura, "Temperature dependence of the viscosity of molten silicon measured by the oscillating cup method," *Japanese Journal of Applied Physics*, vol. 34, no. 7R, pp. 3432–3436, 1995, doi: 10.1143/JJAP.34.3432.

- [98] Okhotin, A. S., A. S. Pushkarskii, and V. V. Gorbachev, *Thermophysical Properties of Semiconductors*, Moscow, "Atom" Publ. House, 1972, (in Russian)
- [99] Hu Zhongxiang, Zhu Lei, Teng Jiaxu, Ma Xuehong, Shi Xiaojun, Evaluation of three-dimensional surface roughness parameters based on digital image processing, *Int J Adv Manuf Technol*, 2008
- [100] ISO 4288:1996, Geometrical Product Specifications (GPS) -- Surface texture: Profile method -- Rules and procedures for the assessment of surface texture