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Controlling Surface Defects of Cold-Sprayed Parts Manufactured on Reusable Tooling

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NOTICES

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14. ABSTRACT
Cold spray additive manufacturing is currently being investigated to produce rotor blade erosion caps. The erosion caps are produced by building a coating on a mandrel that then must be released. Factors influencing the presence of pit defects that occur on the release surface of the coatings are studied in this report. Substrate temperature, material type, and surface roughness are the variables of interest. Substrate (mandrel) temperature can be controlled to reduce pit defect severity and prevent premature delamination in niobium coatings that are produced on hard steel. Mandrel type and surface roughness were also identified as major variables controlling pit defect presence and premature delamination. Future studies are suggested to investigate the aging properties of mandrel materials and to determine appropriate surface pretreatments before a mandrel is used for production.

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cold spray, additive manufacturing, rotor blade erosion caps, mandrel release, reusable tooling, Sciences of Extreme Materials

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1. Introduction

Cold spray additive manufacturing is currently being investigated for use in manufacturing erosion caps for airfoil leading edges. In this work, prototype parts have been produced and robotic equipment has been configured to manufacture erosion caps up to 10 ft long.[1] Niobium (Nb) powder is deposited onto a mandrel to produce a shape that conforms to a given airfoil. After applying multiple layers of the Nb coating, the airfoil cap is removed from the mandrel through mechanical separation using wedges to initiate release at the periphery of the built part. It is critical that the coating is well adhered to the mandrel during the process such that the airfoil does not release prematurely, but not so well adhered that it is exceedingly difficult or impossible to release from the mold.

Balancing "releasability" and adhesion strength can be achieved by applying an initial thin "bond coat" to the mandrel that is sprayed at a lower velocity spray condition than the remaining layers of the part. With this bond coat, a separate issue arises in which localized delamination from the mandrel results in the formation of pit defects across the mandrel. Figure 1 depicts two pieces of Nb material the were applied to a mandrel and release. In one case, the pit defects are rampant, resulting in coating defects that propagate to the surface.

To minimize the pitting defects, preheating the mandrel has been demonstrated as a possible solution. In practice, it has been achieved by spraying hot gas from a cold spray gun onto the mandrel without powder feed. However, no controlled studies of plate temperature (T_{plate}) have been performed to date. This report is a bridge to the gap in understanding how preheat temperature affects pit defects and premature release before the spray process is complete. In addition to temperature, the plate hardness, type, and cold spray condition of the bond coat were also studied. Custom copper hot plates were manufactured by distributing cartridge heaters in copper plates, which enabled consistent and even preheating of various substrate sizes. Four groups of experiments were conducted, and the main outcomes were adhesion strength quantification, a qualitative observation of the severity of pit defects, and determining whether premature delamination occurred. The initial themes of each experiment group are summarized as follows:

1) Small 440C steel substrates of three different hardnesses (42, 48, and 51 hardness on the Rockwell C scale [HRC]) were prepared for coating with Nb to investigate the trends of pit formation with different hardness and starting temperature combinations.
2) Mid-sized 440C steel substrates with three hardnesses (43, 50, and 54 HRC) were prepared for applying single layers of two different Nb bond coat

conditions: a “low-velocity bond coat (LVBC)” and a “medium-velocity bond coat (MVBC).”

3) A 6- × 8- × 0.5-inch plate of 440C steel hardened to 57 HRC served as a substrate for scaled-up test coupons. A smaller strip of similar hardness 440C was prepared with different surface treatments to demonstrate the suspected effect of surface roughness on pit defect severity.
4) Mid-sized 440C and 4340 steel substrates were prepared to 1) assess the viability of material type for mandrel production and 2) identify the impact of repeat occurrences of pit defects.

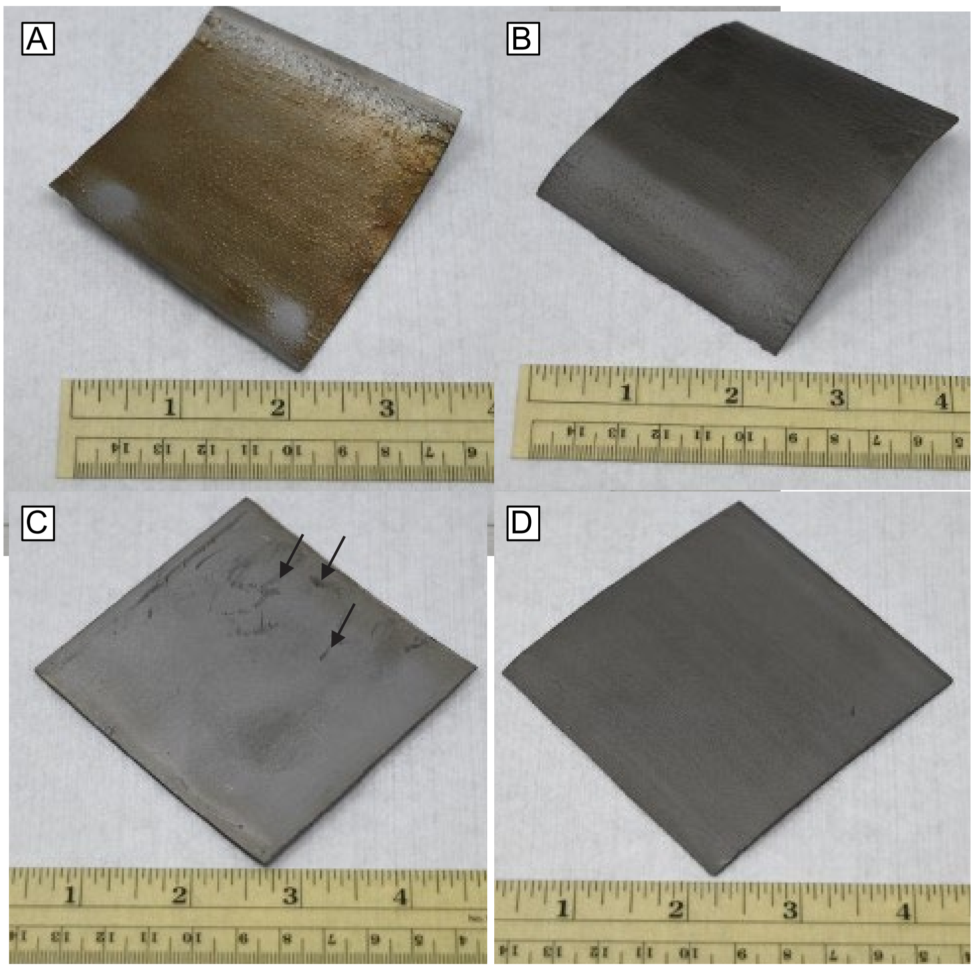

Figure 1. Partial leading edge shapes formed by Nb cold spray that exhibit spray surface-mandrel interface pit defects (A) that propagate to the surface of the spray (B). Discoloraton on the pitted sample is also evident. Similar shapes that show less pitting defects on mandrel contact surfaces (C). Scuff marks are from a wedge used to initiate part release, some of which are indicated by the arrows. Top surface of a released part with minimal defects (D).

2. Methods and Materials

2.1 General Methods and Materials

The Nb powder used for all cold spray conditions was HiT-Nb-A1 powder, lot 000525B043. This powder was procured from Powders on Demand (Solvus Global). VRC Metal Systems GEN III and GEN IV cold spray systems were used for deposition. The system used for each set of experiments and the conditions used for each spray are listed in Section 2.2. For the GEN III, a nozzle no. 58 and block-style applicator were used for all cases. A nozzle no. 96 and standard applicator heater were used with the GEN IV system. All bond coat and topcoat conditions used helium (He) as the process and powder carrier gas. All standoff distances from the nozzle tip to the substrate were set to 1 inch. The specified substrate plate preheat temperatures in the following sections refer to the surface temperature of the plates before applying the "bond coat"—or first layer of deposition—followed by subsequent applications of "topcoat" to build material thickness. For the topcoat conditions, the T_{plate} was not strictly controlled and rose above the initial preheat temperature due to heat imparted to the substrate by the hot spray gas.

Line roughness measurements (Ras) were taken using a Mitutoyo SJ-210 profilometer using a scan rate of 0.02 inch/s. Optical measurements were performed using either a Keyence or Olympus microscope and are discussed in the section each was first used.

2.2 Specifics for Each Set of Experiments

2.2.1 Preliminary Coupons: Experiment Set 1

The first experiment set comprised small 440C stainless steel plates that were heat treated to three hardness levels. The plates' dimensions were approximately 2 × 2 × 0.5 inches. A hot plate was constructed from a piece of 1/4-inch-thick copper plate and three insertion heaters (Figure 2A). A thermocouple inserted in the copper plate was used for temperature control (TcC). Preliminary experiments were performed to determine the approximate offset of plate surface temperature (measured by Tc1, Tc2, Tc3) from TcC. An example of the heating characteristics of the plate are shown in Figure 2B, which demonstrates equivalent heating of each plate and an approximate temperature offset of 10 °C. This value was not constant for a given setpoint temperature and was determined independently for each experiment.

Figure 2 shows the 440C steel plates arranged on the hot plate. Samples were sprayed toward the bottom of each plate. The numbered portions in the figure are explained as follows:

1) Light gray surface roughened from a previous application of material.
2) Surface of a plate not sprayed.
3) Thermocouple locations tested. Ultimately, the thermocouple used to measure surface temperature was taped to the front surface of the plate that was sprayed.
4) Steel shim stock was positioned between the substrates and the hot plate to protect the hot plate from overspray material.
5) Fiberglass tape was placed between the hot plate and an aluminum support used to support the setup.

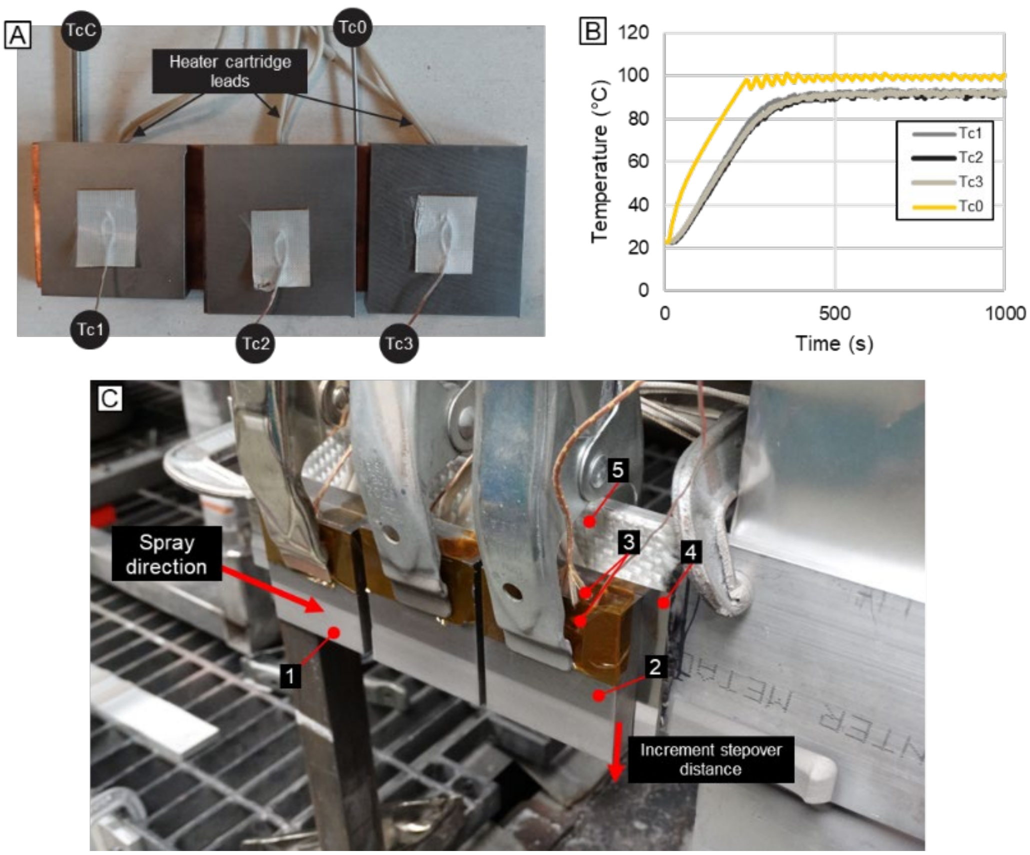

Figure 2. (A) Different hardnesses of 440C steel plates arranged on a custom hotplate for initial heat-up testing. (B) Initial heat-up testing of the three plates. (C) Photograph of the 440C substrates mounted to a hot plate in position for the cold spray experiments.

A VRC Metal Systems GEN III high-pressure, cold spray system was used for this set of experiments. The general spray conditions are outlined in Table 1. The LVBC conditions for the bond coat are outlined here.

Table 1. **Spray conditions for each sample prepared in the initial heated plate trials.**

Parameter	Bond coat	Topcoat
Spray system pressure (psi)	400	450
Spray system temperature (°C)	550	600
Carrier gas flow rate (SLM)	150	110
Powder feeder rpm	3.5	8
Raster speed (mm/s)	400	500
Indexing (mm)	0.5	0.5
Layers applied	1	. . .

Raster speed is defined as the traverse speed of the spray gun across the substrate, and indexing is the distance between each parallel line traversed by the spray gun. The variation in condition between each sample sprayed is captured in Table 2. After the first three plates, the topcoat was changed from three to six passes to build additional deposit thickness for easier release. The preheating temperature of the plate surface before a spray is defined as the T_{plate}.

Table 2. **Variation between parameters for each sample number.**

Sample	Plate hardness (HRC)	T_{plate} °C	Topcoat layers
1	42	22	3
2	48	22	3
3	51	22	3
4	42	100	6
5	48	100	6
6	51	100	6
7	42	150	6
8	48	150	6
9	51	150	6

2.2.2 Dolly Adhesion Testing: Experiment Set 2

The second set of experiments contained larger plates than the initial experiments, and only a single layer of the bond coat condition was sprayed. The 440C substrates were preheated to hardnesses of 43, 50, and 54 HRC. Figure 3 depicts the three substrates mounted to a copper hot plate. The hot plate comprised four 300-W cartridge insertion heaters inside of a 6- × 8- × 0.5-inch copper plate. The plate was designed such that substrates could be clamped on to it or secured with bolts as done in Section 2.2.3. Similar to experiment set 1, thermocouples were affixed to the face of each plate (Tc1, Tc2, and Tc3), with a thermocouple inserted in the hot plate for TcC. Tc0 was attached to a data logger to record the hot T_{plate}, and it is interchangeable with TcC. Details for setting the surface temperature for each experiment are omitted but followed the same strategy as the prior experiment set. Variables that remained constant for this set of experiments are summarized in Table 3.

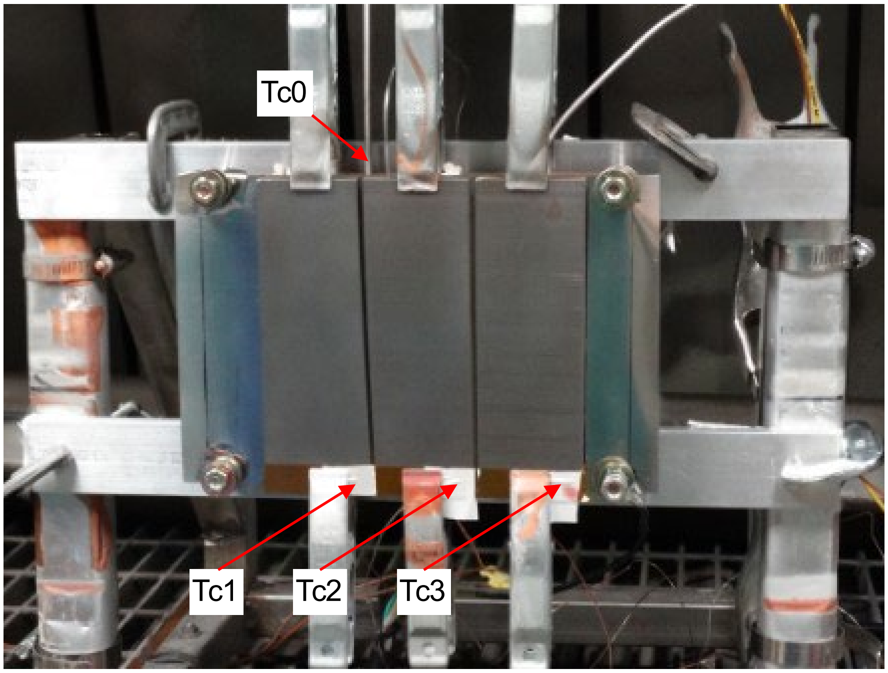

Figure 3. Heated 440C substate arrangement to produce films for adhesion testing.

Table 3. Variables held constant for each sample prepared for adhesion testing.

Parameter	Bond coat
Carrier gas flow rate (SLM)	150
Powder feeder rpm	3.5
Raster speed (mm/s)	400
Indexing (mm)	0.5
Lines of material per layer	254
Layers applied	1

Table 4 gives the variables specific to each sample produced. Samples are coded in the format N-M, where N is the order in which the spray was performed and M denotes the plate hardness (1, 2, 3 correspond to 43, 50, and 54 HRC). Each plate hardness was sprayed simultaneously using the apparatus in Figure 3 (i.e., 3-1 is the sample sprayed on the 43 HRC plate from the third spray performed. It was sprayed at the same time as 3-2 and 3-3). Spray system pressure and temperature are abbreviated as P_{spray} and T_{spray}, respectively. Two different bond coat conditions–LVBC and MVBC–were evaluated. The MVBC is the 425 psi, 580 °C spray condition.

Table 4. Experimental parameters varied between each sample.

Sample	T_{plate} (°C)	P_{spray} (psi)	T_{spray} (°C)	Plate hardness (HRC)
1-1	150	425	580	43
1-2	150	425	580	50
1-3	150	425	580	54
2-1	20	400	550	43
2-2	20	400	550	50
2-3	20	400	550	54
3-1	100	400	550	43
3-2	100	400	550	50
3-3	100	400	550	54
4-1	75	400	550	43
4-2	75	400	550	50
4-3	75	400	550	54
5-1	22	425	580	43
5-2	22	425	580	50
5-3	22	425	580	54
6-1	75	425	580	43
6-2	75	425	580	50
6-3	75	425	580	54

Gouging of some plates occurred between some sprays due to the difficulty of removing the single layer, soft material. Thus, the plates were refinished with a face mill after two of the spray conditions: samples 1-M and 3-M. All samples were prepared with Scotch-Brite and cleaned with alcohol before each spray.

Adhesion testing was performed in accordance with ASTM D4541.[2] Adhesion testing dollies were glued to each coating using EP11HT adhesive. Excess adhesive was trimmed from the dolly edges, then the adhered samples were cured in an oven set to 100 °C for 1 h. Samples were allowed to cool to room temperature before adhesion testing with a DeFelsko PosiTest automatic adhesion tester. The coating was not trimmed around the edges of the dollies for any test after curing the adhesive. A pull rate of 300 psi/s was used for the 14- and 10-mm dollies.

Profilometry of the dolly faces was performed with a Keyence VK-X1000 to investigate trends in area surface roughness (Sa). For the measurements of dolly surfaces, a waveform surface shape filtering was applied to each sample as well as a 200-µm S filter.

2.2.3 Scale-Up of Findings: Experiment Set 3

Experiment set 3 had the same hot plate as the prior set, but instead of separate substrates, a single 6- × 8- × 0.5-inch plate of 57 HRC 440C steel was used. The arrangement is depicted in Figure 4A and B. The plate was prepared before spraying by surface grinding to Ra values of 18.1 microinches (µin) measured colinearly to the 6-inch dimension and Ra values of 7.8 µin in the long dimension

(Figure 4C). In this set of experiments, the VRC GEN IV cold spray system was used. The variables held constant between each spray are summarized in Table 5.

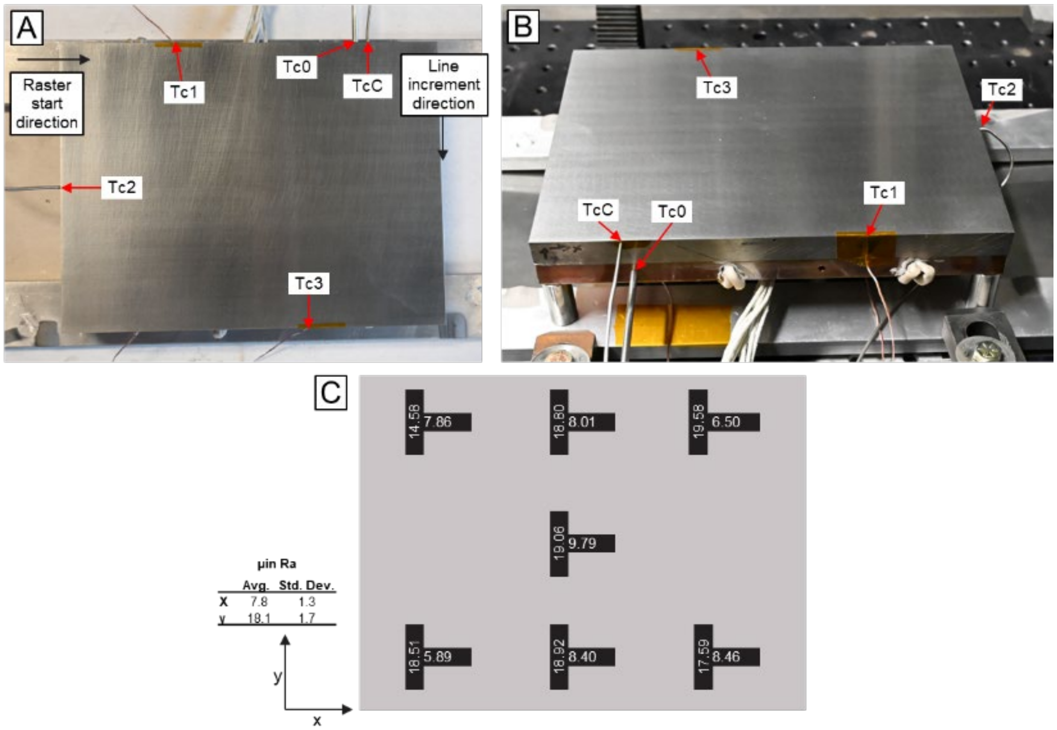

Figure 4. Large 440C substrate arrangement showing the thermocouple locations and spray directions (A). Alternate angle (B) of the plate showing the control thermocouple (TcC) inserted in the substrate and thermocouple used for data logging the hot T_{plate} (Tc0). Surface Ra at various locations measured with a stylus profilometer and reported in µin Ra (C).

Table 5. Parameters held constant for each sample prepared on the large 440C substrate.

Parameter	Bond coat	Topcoat
Spray pressure (psi)	. . .	450
Spray temperature (°C)	. . .	600
Carrier gas flow rate (SLM)	. . .	60
Powder feeder rpm	3.5	. . .
Raster speed	400	. . .
Indexing	. . .	0.5
Lines of material per layer	. . .	. . .
Layers applied	1	. . .

Table 6 shows the variation of parameters between each condition. The change from 500 to 400 mm/s raster speed was made to streamline the experimental procedure. The topcoat parameters used a higher-powder feed rate to build up total thickness faster and conserve He gas.

Table 6. Variation in parameters between samples prepared on the large 440C substrate.

Sample	Bond coat						Topcoat		Bond and topcoat
	T_{plate} (°C)	P_{spray} (psi)	T_{spray} (°C)	Indexing (mm)	Carrier gas flow (SLM)	Raster speed (mm/s)	Powder feeder rpm	Layers	Lines of material/layer
1	100	400	510	0.5	60	500	8	<8[a]	268
2	100	400	550	0.5	150	400	12	4	50
3	100	425	580	0.5	150	400	12	4	50
4	22	400	550	0.5	150	400	12	4	50
5	100	425	580	3.1	150	400	12	4	50
6	150	400	550	0.5	150	400	12	4	50
7	22	425	580	3.1	150	400	12	4	50

[a] The spray was stopped during the application of an eighth topcoat layer.

An Olympus OLS5100 laser scanning microscope with a 5× objective was used to image the surface of the strip deposits and determine Sa. A total of 35 images were stitched for each sample. Noise reduction and polynomial shape correction were applied in postprocessing. Due to a high degree of warping, it was difficult to establish a flat baseline for the partially curved surfaces, particularly in sample 4.

A separate experiment was performed on an approximately 57.5 HRC 400C stainless steel strip with a dimension of 8 × 1.6 × 0.5 inch. The surface was modified before applying a layer of the MVBC and topcoats, in alignment with the methods of the large plate sprays (sample 3 from Table 6). One area of the plate was treated only with Scotch-Brite, whereas another was treated by grit blasting with 60-grit, silicon carbide blasting media in a Trinco Dry Blast Grit blasting chamber with a gun pressure of 70 psi. The third section was treated with a low-velocity Nb spray condition. This treatment is referred to as "powder blasting" and comprised a single-layer spray condition operating at 650 psi, 125 °C, 150 SLM of powder feed gas, 3.5 rpm of powder feed, and nitrogen gas. This condition was also treated with Scotch-Brite before spraying to remove loose particles.

2.2.4 Comparison of Mandrel Materials and Surface Condition: Experiment Set 4

The substrate heating arrangement depicted in Figure 3 was used again in this experiment set to prepare deposits on substrate plates of varying hardness. However, adhesion testing was not performed and only the released surface was studied. Three plates of varying hardness were prepared from both 440C stainless steel and 4340 steel. The plates were cleaned with alcohol, Scotch-Brite, and then a final alcohol rinse before each spray. Ra using a stylus profilometer were taken of the substrate surface before each spray, after each spray, and after each surface preparation.

Cold spray parameters remained constant for each plate sprayed, except for the amount of raster lines of spray over the surface. The general conditions used while spraying with the VRC GEN IV system are given in Table 7, and the variations of parameters between each spray are given in Table 8.

Table 1. Spray parameters used for the bond coat and topcoat.

Parameter	Bond coat	Topcoat
T_{plate} (°C)	100	. . .
Spray pressure (psi)	425	450
Spray temperature (°C)	580	600
Carrier gas flow rate (SLM)	150	60
Powder feeder rpm	3.5	12
Raster speed (mm/s)	400	400
Indexing (mm)	0.5	0.5
Layers applied	1	4

Table 2. Parameters varied for each sample prepared to study substrate type and surface condition.

Sample	Plate hardness (HRC)	Plate material	Raster lines
1-1	43	440C	50
1-2	50	440C	50
1-3	54	440C	50
2-1	43	440C	100
2-2	50	440C	100
2-3	54	440C	100
3-1	44	4340	50
3-2	50	4340	50
3-3	54	4340	50
4-1	44	4340	100
4-2	50	4340	100
4-3	54	4340	100

3. Results and Discussion

3.1 Preliminary Evaluation: Experiment Set 1

This set of experiments served to test the viability of further substrate preheat studies. Photographs of the delamination surface of Nb deposits sprayed on 440C steel plates of varying surface temperatures and hardnesses are depicted in Figure 5.

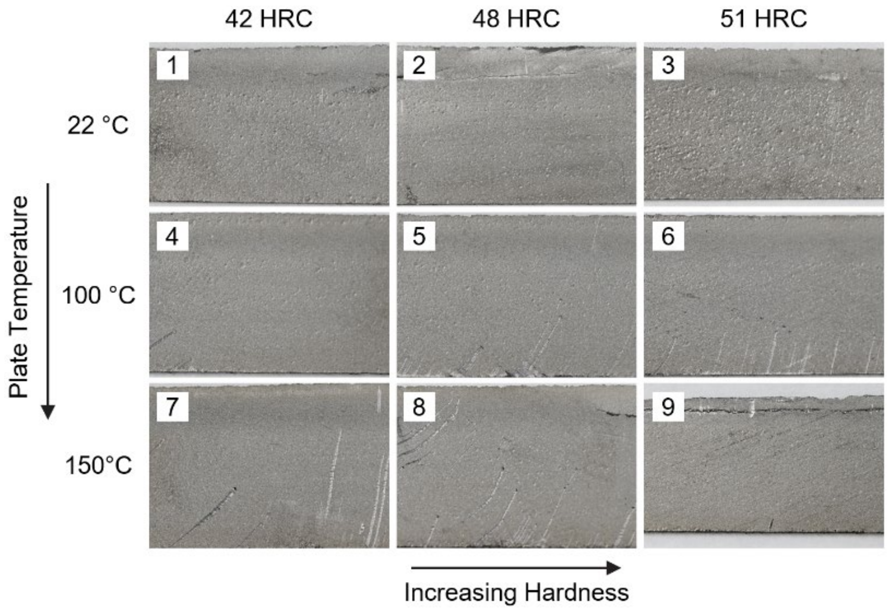

Figure 5. Surfaces of the Nb deposit that were adhered to and released from the small substrates of 440C steel with varying hardnesses and surface temperatures.

The appearance of pitting defects appears to decrease with increasing T_{plate} and decreasing plate hardness. Qualitatively, samples were more difficult to release on the lower hardness plate and at higher temperatures. This set of experiments encouraged a further investigation into the adhesion strength of the coatings as a function of hardness and substrate temperature. As discussed in Sections 3.3 and 3.4, the presence of pit defects is also attributed to the beginning surface roughness of the substrates and was not accounted for in these preliminary experiments.

3.2 Adhesion and Surface Defect Investigation: Experiment Set 2

The adhesion testing performed on the substrates of varying temperature/hardness are summarized in Figure 6. Refer to Section 2.2.2 for an explanation of sample labeling.

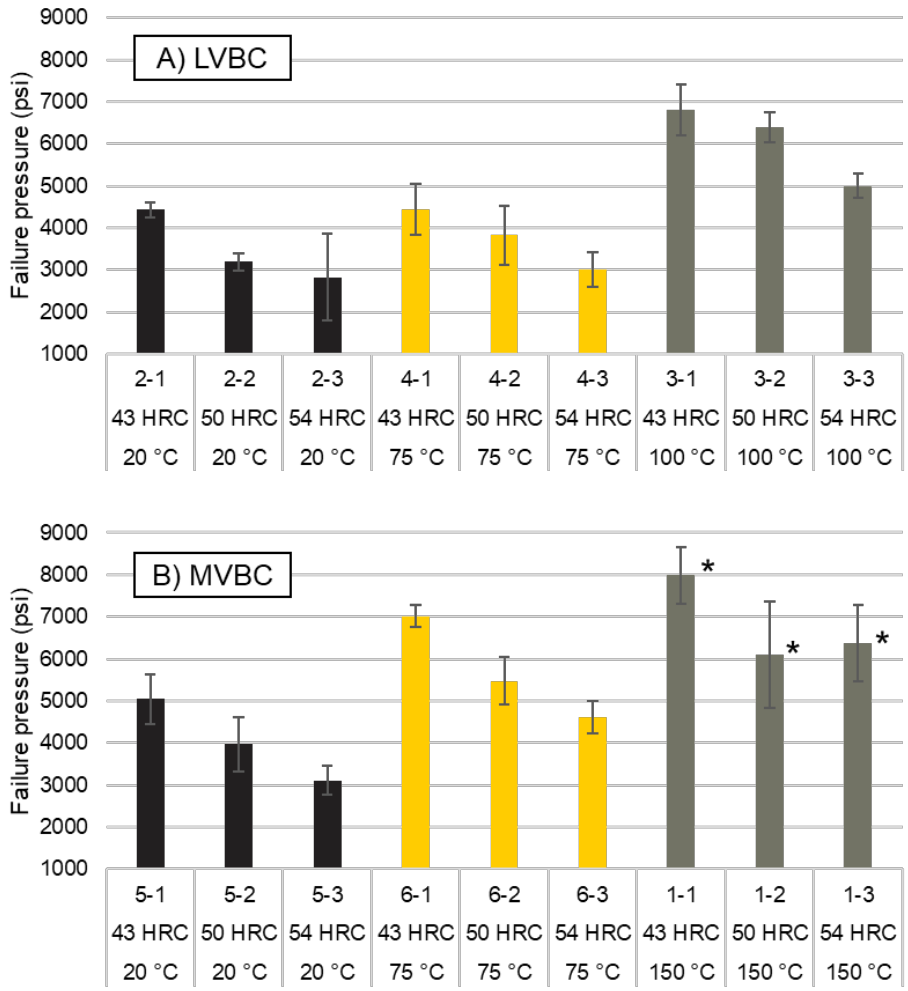

Figure 6. **Adhesion testing of various combinations of bond coat condition (A, LVBC or B, MVBV), substrate hardness, and substrate temperature. Labels below each bar indicate the sample identifier, plate hardness, and plate preheat temperature from top to bottom. *Testing was performed with 10-mm dollies in the 1-M samples because the adhesion strength was close to, and in some cases exceeded, the limit of 14-mm dollies.**

For all samples except the MVBC samples on 150 °C substrates, the adhesion strength decreased with increasing hardness and increased with increasing T_{plate}. The 10-mm dollies had to be used because the maximum measurable adhesion strength with 14-mm dollies was reached on the 1-M samples. Adhesive was more difficult to trim from the dolly edges on the 10-mm samples and likely influenced the error in the results of 1-N samples. In addition, the higher-condition MVBC resulted in higher adhesion strengths than the LVBC. Two datapoints from the 3-1 sample did not fail within the limits of the adhesion test (7143 psi), and thus the value was simply recorded as the maximum measurement observed.

It is evident that adhesion strength can be controlled by adjusting the plate preheat temperature, hardness, and bond coat spray condition. However, the presence of pit defects seems to be a more complex issue. Pitting defect severity tended to vary based on the area sampled. Photographs of the released surface obtained during dolly pull experiments are summarized in Figure A-1 in the Appendix, which depicts the variation in pit defects. Additional images were obtained using a laser microscope to identify pitting on the as-released surfaces. Representative images are summarized for the LVBC in Figure A-2 and MVBC in Figure A-3 in the Appendix. There is no clear, qualitative trend of pit defect presence between plate preheat temperatures. However, the amount of pitting does appear to be less severe on the low hardness substrates. Pitting defects were suspected to also be a function of the substrate surface condition, as well as the surface temperature. Surface condition is explored in Sections 3.3 and 3.4 because it was not explicitly controlled in this set of experiments.

For substrate temperatures, well-controlled experiments with identical surface treatment between sprays should be considered. The approach herein of spraying a substrate, releasing the coating, then machining if necessary does not result in constant substrate condition between coating applications. Within a spray condition, however, the surface condition can be treated as near constant across a given substrate. Figure 7A depicts a sprayed substrate after four dolly samples were pulled. The robot raster path began above dolly A, and with each additional raster of the robot it progressed toward dolly D. During this process, the substrate heats up above its initial preheat temperature. Therefore, the substrate was at the highest temperature after dolly D was sprayed and lowest before dolly A was sprayed. The difference in Sa was investigated between dolly A and dolly C. The Sa was measured using a Keyence laser microscope. A box and whisker plot of the difference in Sa and pit depth (area below the average height of the surface) are given in Figure 7B and C, respectively. Figure 7B shows that, on average, dolly A has a higher Sa than dolly C. In addition, a similar indication is apparent when observing the total pit depth difference. The pit depth is measured as a negative value (i.e., the total pit depth, or sum of depth of all the measured pits, is an amount below the baseline height). Since the average difference in pit depth in Figure 7 is negative, it can be inferred that, on average, dolly A has an average pit depth that is deeper than dolly C. Hence, the continual increase in T_{plate} during the spray results in a defect gradient across the substrate.

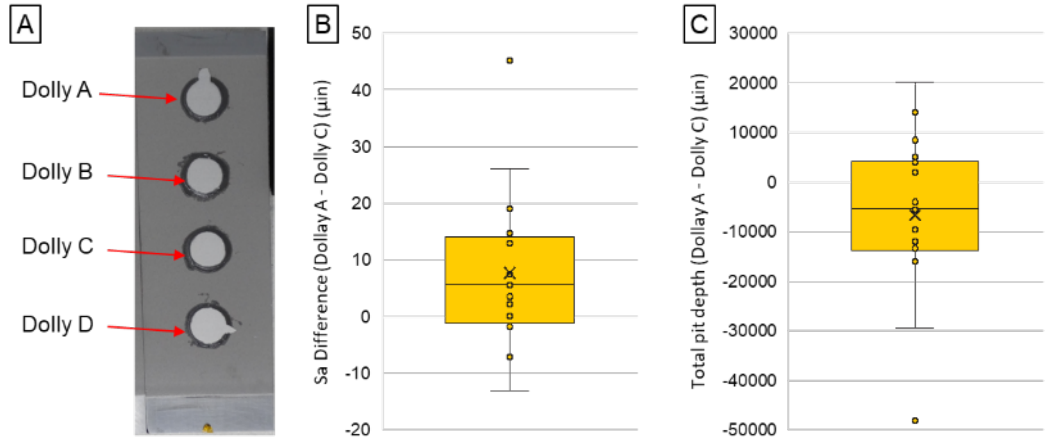

Figure 7. **(A) Dolly location as pulled from a coating after adhesion testing. (B) The difference in Sa as measured by a Keyence laser microscope between dolly A and dolly C. (C) The difference in the total depth of all pits measured between dolly A and dolly C.**

3.3 Large 440 C Plate Substrate Tests: Experiment Set 3

A large area 440C steel plate was used to spray coatings that could be released and examined for pit defects. The first spray used most of the plate area and served to investigate thermal characteristics during coating application, as well as investigate whether preheat alone would successfully control pitting defects. The arrangement of the plate and delaminated coating are shown in Figure 8A and B, respectively. Two thermocouples (Tc1 and Tc3) were mounted to the edges of the plate with epoxy, and another was inserted into the edge of the plate. The temperatures measured during preparation of sample 1 are plotted in Figure 8C.

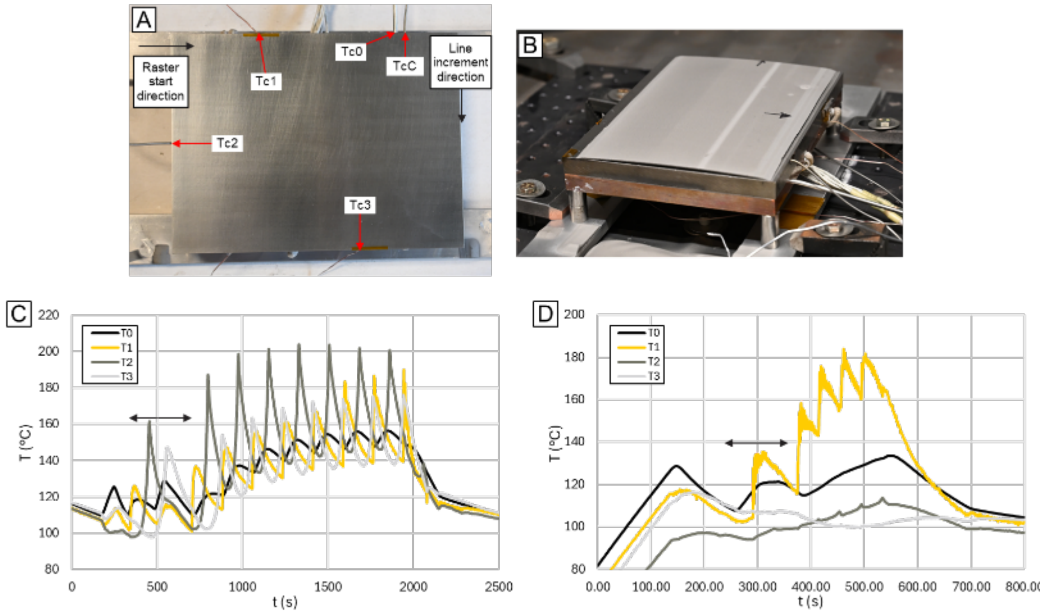

Figure 8. (A) Substrate arrangement showing spray direction and thermocouple locations. (B) Delaminated plate (sample 1) resting on the substrate. (C) Thermocouple measurements from preparing sample 1. (D) Thermocouple measurements obtained while preparing a smaller sample (sample 2). The arrows in C and D depict the approximate time span of the bond coat application.

As the spray path of sample 1 progressed from Tc0 toward Tc3, the thermocouples show an increasing temperature response, indicating a periodic increase in T_{plate} from the spray process. Tc2 rises in a more exaggerated manner due to its parallel positioning to the raster direction. During the spray, hot gas and particles directly impact Tc2 but not Tc1 or Tc3. Heat accumulation during the spray is from all thermocouples including Tc0. Tc0 reflects the temperature of the copper hot plate used for TcC. Although active heating was needed for preheating the plate, the temperature rise above the 120 °C setpoint caused the controller to stop applying power to the hot plate once the spray progressed past the bond coat application. Although sample 1 successfully bonded to the plate, it exhibited considerable amounts of pitting defects. Thus, a series of sprays were conducted over a smaller area of the plate to further understand the importance of certain process parameters on the formation of pit defects while conserving powder and He gas. Figure 8C depicts the thermal history of the plate when sample 2 was sprayed over a smaller area. The temperature rise is mostly apparent in Tc1, indicating that substrate heating from the hot spray gas is a local phenomenon.

Sample 1 was prepared using a modified version of the LVBC method, because it was the first sample sprayed using the new GEN IV cold spray system. The carrier gas flow rate was reduced because the GEN IV system allows lower flow rates compared with the GEN III system. To maintain a similar carrier gas/hot gas mixture temperature to that used with the GEN III system, the process temperature was adjusted. However, significant pitting defects were observed on Sample 1. Consequently, the system settings were returned to the original parameters to ensure consistency with previous experiments. A series of sprays were conducted over a smaller area of the plate to further understand the importance of certain process parameters on the formation of pit defects while conserving powder and He gas. A photograph of a sample applied to the plate in the process of being removed with a wedge is given in Figure 9. The delamination surfaces for each deposit are shown in Figure 10.

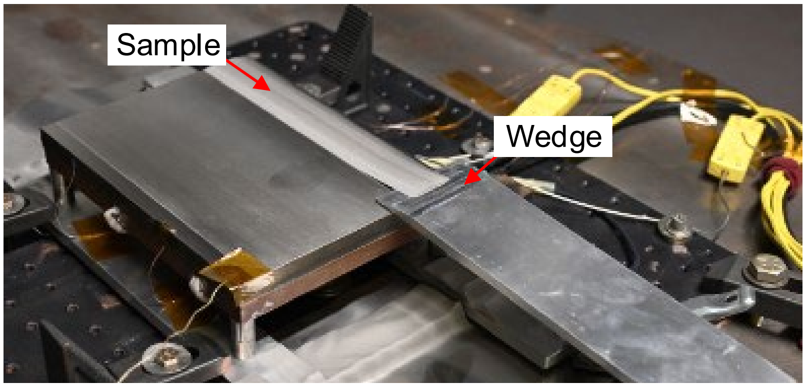

Figure 9. A smaller strip deposit being removed from the large 440C substrate using an aluminum wedge tool.

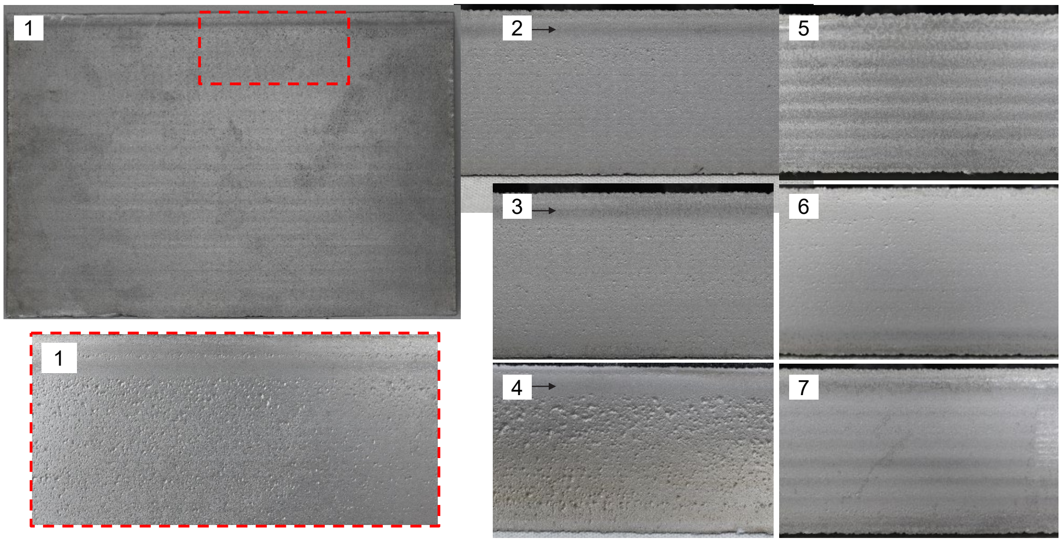

Figure 10. The surface of each numbered sample after release from the substrate. For scale, samples 2–7 are approximately 30 mm from top to bottom while plate 1 is 140 × 205 mm. The arrows in samples 2–4 indicate the smooth surface of the first line of spray material deposited.

Samples 2 and 3 differed by the bond coat condition used: LVBC versus MVBC. Photographs of sample 3 indicate slightly less severe pitting than sample 2. Sample 4 can be compared with sample 2 to showcase the importance of substrate preheating temperature. Sample 4 exhibited a concave surface and severe pitting on the delaminated face, unlike the surface of sample 2. The concave surface suggests that some delamination occurred during the spray. In this case, the deposit would be anchored to the substrate at the top and bottom edges while the center portion becomes detached. Visually, there is little difference in pit defects between samples 2 and 6 where the preheating temperature was changed from 100 to 150 °C, indicating that there is some temperature threshold in which a further increase had diminishing returns on the presence of pitting defects. We concluded from these observations that preheating the substrate above room temperature is a major factor in the delaminated surface quality. In this case, because coatings were applied in the same area multiple times, and the entire plate was covered when preparing sample 1, the substrate effect of Sa was assumed to be a minimal factor contributing to the presence of pit defects on samples 2–7.

Height maps of the deposit release surfaces to compliment Figure 10 are given in Figure 11, along with the area roughness (Sa). It is evident that the pit defects were more extreme in sample 4, which was not sprayed with substrate preheating. In addition, the large-index, distance bond coats (5 and 7) exhibit the lowest Sa and degrees of pit defects. The profilometry was conducted over an area representative of the general deposit; however, there is some variation across each strip. For example, sample 3 appears to have less severe pitting than sample 2 in Figure 10A, whereas the area sampled in Figure 11 indicates that sample 3 is rougher than sample 2.

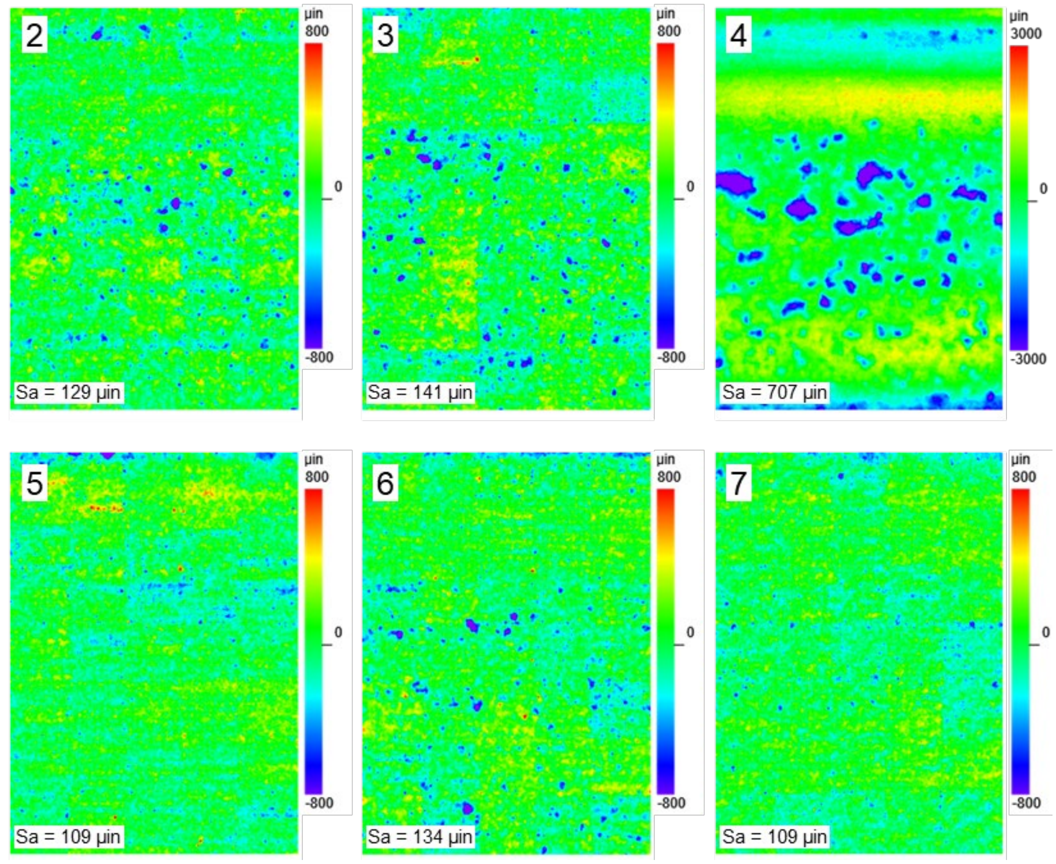

Figure 11. Height map of a 0.646- × 0.464-inch area of each sample, with the Sa roughness measurement overlaid. The top edge in each sample appears to have the most consistent surface finish and is the initial line of material deposited by the spray gun. The high spots indicated may be attributed to reflections from the material rather than maxima. Profilometry images were corrected to improve general flatness and therefore should not be taken as an exact representation of the bulk topography.

At the start of the bond coat application, the first line of spray in each sample left a near-pristine surface without pitting defects, even on sample 4, which had the most severe pitting. The sample labels 2, 3, and 4 in Figure 10 overlay the first line of bond coat applied, which appears as a horizontal line from left to right across the deposit. This observation motivated the increase of stepover distance between raster lines of coating application from 0.5 to 3.1 mm such that each spray line of the bond coat had little overlap with the line before it. For reference, the spray plume width is approximately 5 mm, which is as a good reference value for the diameter of the spray plume. Samples 5 and 7 were sprayed with the 3.1-mm stepover. They displayed few pit defects, and if the defects were present, they occurred away from the center of the raster pattern in the area where the overlap between lines occurred. The Ra of the substrate in Figure 12A demonstrates that the area of bond coat application influences the local substrate Sa. Pitting locations are identified between the raster lines in Figure 12B, and the local Ra on and between raster lines is given in Figure 12C. Samples 5 and 7 were also more difficult to release from the substrate than samples 2, 3, and 6. This increased stepover approach may be useful provided a non-preheated substrate is required, because sample 7 was prepared with a room temperature substrate and exhibits limited pit defects.

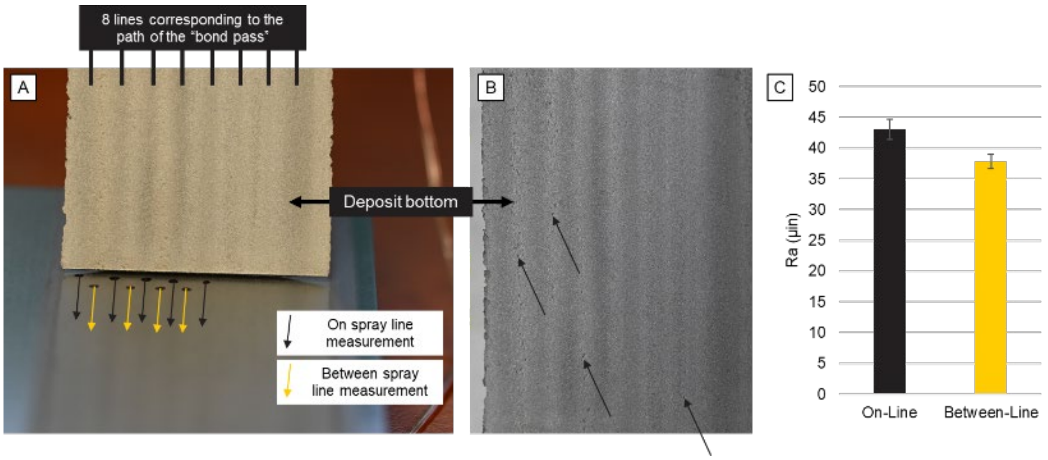

Figure 12. Examination of the substrate roughness (A) and pit defects (arrows in B) on the deposition surface that was released from the mandrel. Substrate Ra (C) from the areas depicted in panel A.

To understand the influence of surface condition before bond coat application, a sample of 440C steel (~57.5 HRC) was prepared with three surface treatments (Figure 13A). One area was prepared by rubbing with Scotch-Brite only, whereas another was pre-sprayed with a low-temperature Nb spray condition, referred to as powder blasting. The third area was roughened with 60 grit blasting media. A bond coat and topcoat of Nb were applied across the entire coupon, as described in Section 2.2.3. Immediately after the spray, the Scotch-Brite-only area and the powder-blasted area were delaminated from the substrate while the grit-blasted area remained firmly attached (Figure 13B).

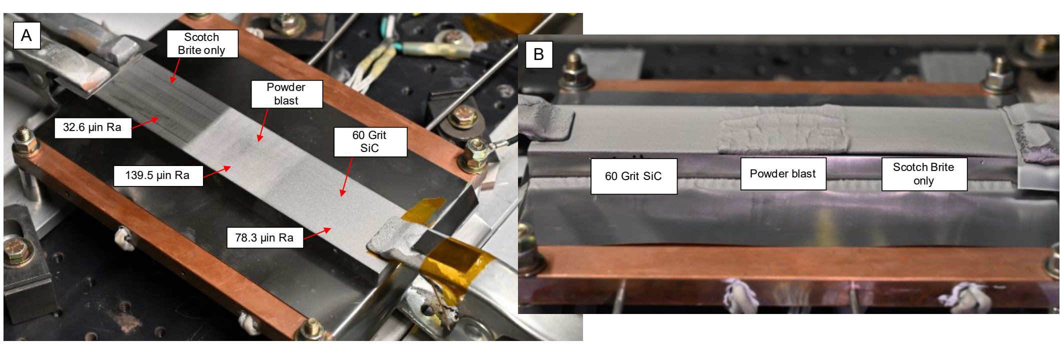

Figure 13. (A) The 440C steel substrate prepared to three separate surface conditions before spraying, and the deposition immediately after termination of the spray (B) showing delamination from the substrate in the powder- and grit-blasted areas.

The top surface and underside of the released deposit are shown in Figures 14A and B, respectively. Severe delamination and wrinkling are evident in the powder blast-only section. In the Scotch-Brite-only section, some pit defects are observed in addition to warping of the deposit. The rough substrate of the grit-blasted area appeared to have fewer pit defects and substantially less warping.

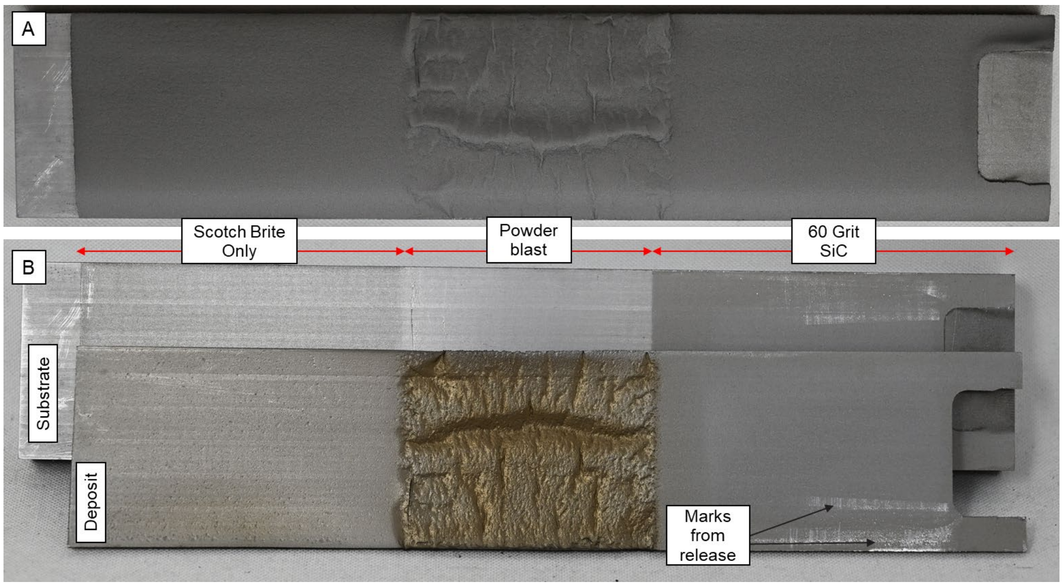

Figure 14. (A) Deposit resting on the substrate before release showing substantial "wrinkling" distortion in the powder-blasted area. (B) The underside of the deposit overlaid on the release surface of the substrate.

Height maps of the delamination surface were acquired using a confocal laser microscope to display the drastic differences in surface quality. The smooth substrate produced a rougher deposit surface and deeper pit defects (Figure 15).

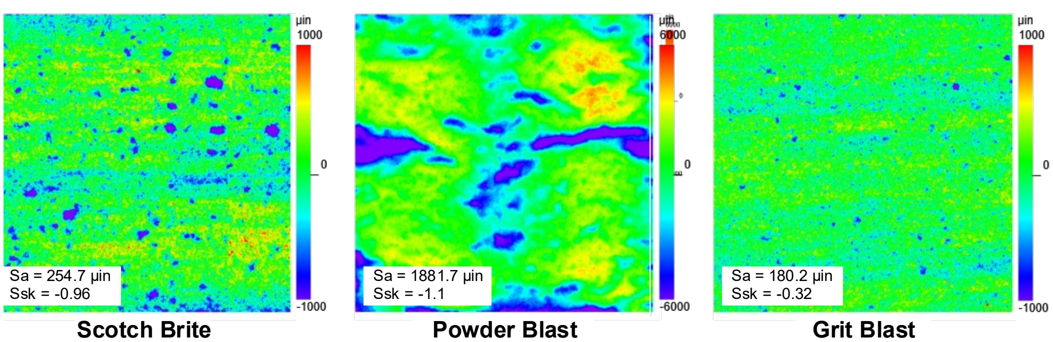

Figure 15. Height maps of the delamination surface from the deposit produced on the 440C stainless steel substate with different surface treatments. Area roughness (Sa) and skewness (Ssk) are overlaid. Dimensions of the images are 0.465 × 0.465 inch.

Measurements of substrate Ra were taken before spraying, after releasing the deposit, and after cleaning the released area with Scotch-Brite. The measurements are summarized in Figure 16. For the powder-blasted area, the roughness was also taken immediately after powder blasting the substrate. The surface was rougher than the other treatments. Preparation with Scotch-Brite before spray application reduced the roughness, suggesting that a loose layer of particles was adhered to the substrate from the powder-blasting condition. This sparse, poorly bonded, initial coating is attributed to the premature release observed during spraying. In addition, the decrease in roughness after the final cleaning step post release indicates that loose particles may remain in the substrate. This observation suggests that cleaning the substrate between spray applications may be necessary for consistent release performance and surface quality. At this time, it is unknown how the surface roughness will change as a substrate is reused in a manufacturing process. This warrants further study on the progression of mandrel surface roughness and morphology, which may include multiple initial surface treatments. Options such as grit blasting to various roughness values, grinding, or as-machined surfaces could be explored. A preliminary investigation of mandrel material aging is presented in Section 3.4.

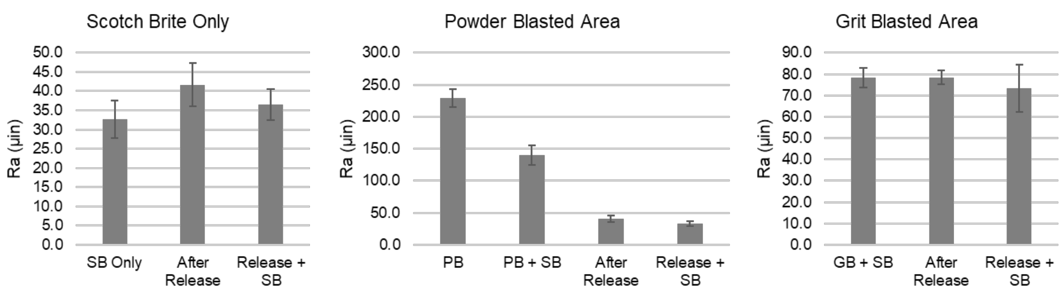

Figure 16. Ra for each section of the substrate plate for a given process step. Definitions for the acronyms are as follows: SB = Scotch-Brite; PB = powder blast; and GB = grit blast. Measurements were taken after the surface preparation step, after release of the Nb deposit, and cleaning with SB after release.

3.4 Material Type Evaluation: Experiment Set 4

Substrates of 440C stainless steel and 4340 steel hardened to similar values were prepared to evaluate the effect of substrate material type on coating surface quality. Each substrate was sprayed twice to evaluate how the substrate material changing between sprays influences the formation of pit defects. The first spray used a narrower spray area (less raster lines) than the second spray. Thus, fresh substrate and substrate area that had been sprayed were both covered during the second spray. Other spray parameters were consistent between each spray, as outlined in Section 2.2.4.

The deposits from the first application of Nb to the 440C substrates are shown in Figure 17. Pitting defects are evident in the plates harder than 43 HRC. Even the surface of coating 1-2 appears to be rougher than 1-1 due to the propagation of the pit defect shape through the material (Figure 17A). Sample 1-3 delaminated before the spray terminated due to poor adhesion and subsequently a high degree of deep pit defects. Delamination occurred during the final layer of material application, and thus part of the sprayed area was coated with a thin layer of topcoat condition material. This material had to be removed by peeling it from the substrate with a razor blade.

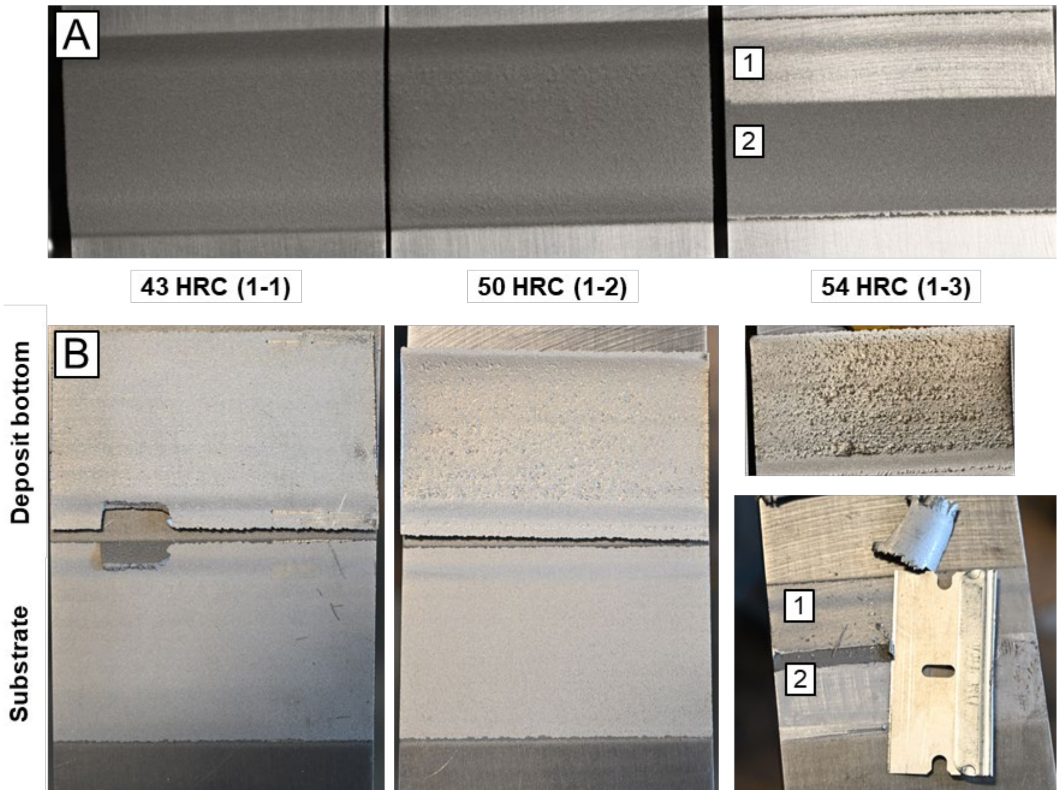

Figure 17. (A) Nb deposited on 440C steel material upon initial application of material. (B) The delaminated deposits from the first spray showing pit defects. Sample 1-3 delaminated toward the end of the spray. Area 1 of sample 1-3 was not sprayed on by the final pass of the topcoat condition, whereas area 2 was sprayed on with the topcoat condition after the deposit delaminated during the spray.

The second set of samples consisted of a coating applied to the released area and non-sprayed area from the first set of samples. Previously sprayed on surfaces did not result in delamination and had a lower degree of pit defect occurrences, as depicted in Figure 18. A drastic decrease in pit defects is evident in area 3b of sample 2-3, and a slight reduction is evident in area 3a compared with the initial spray that delaminated. These results highlight the importance of spray conditions on the Sa change in the substrate. Delamination was not observed on the hard substrate area that was not sprayed on previously in this case. We suspect that the previously sprayed-on area anchored the deposit and prevented delamination. In addition, the fresh substrate area was likely hotter in this case because it was sprayed on after the previously sprayed area.

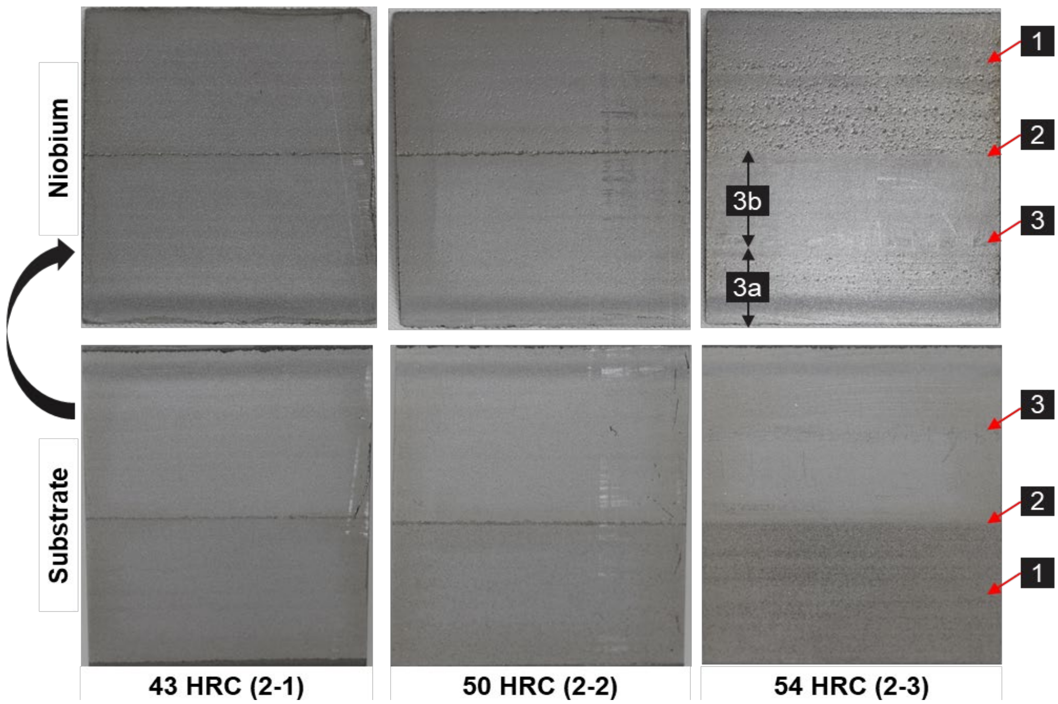

Figure 18. Deposits formed on the 440C substrates on two different areas. One area was previously sprayed on (3), and the other was not sprayed on (1). The separation between the two areas is indicated by 2, red arrow. Area 3a is the area from which the deposit delaminated from in 1-3, and (3b) is the area that was coated with a layer of topcoat after the deposit delaminated during the spray.

The process used for the 440C plates described above was repeated with the heat-treated 4340 steel substrates. The initial spray is depicted in Figure 19, and the second spray is depicted in Figure 20.

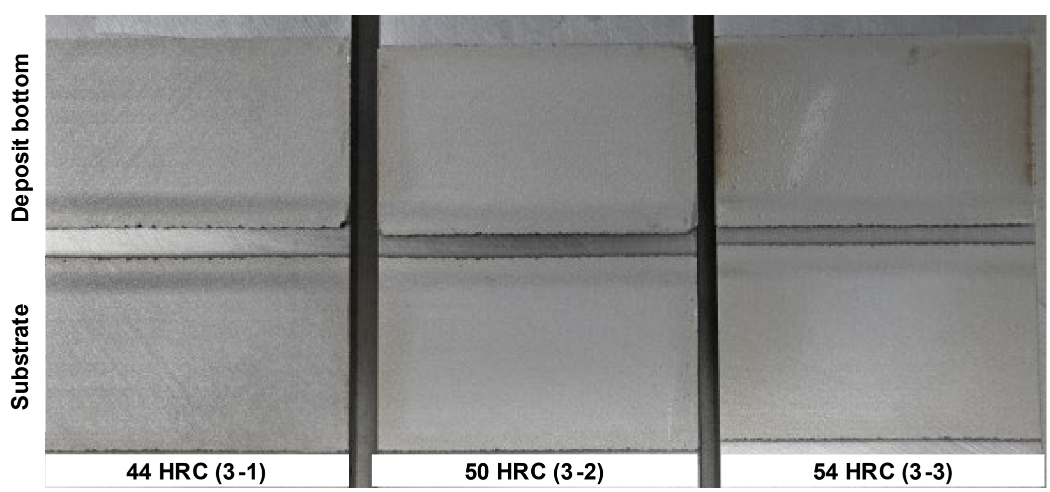

Figure 19. Delamination surfaces of the cold spray deposits sprayed on 4340 steel substrates, flipped over, and resting next to the area that they were released from on the substrate.

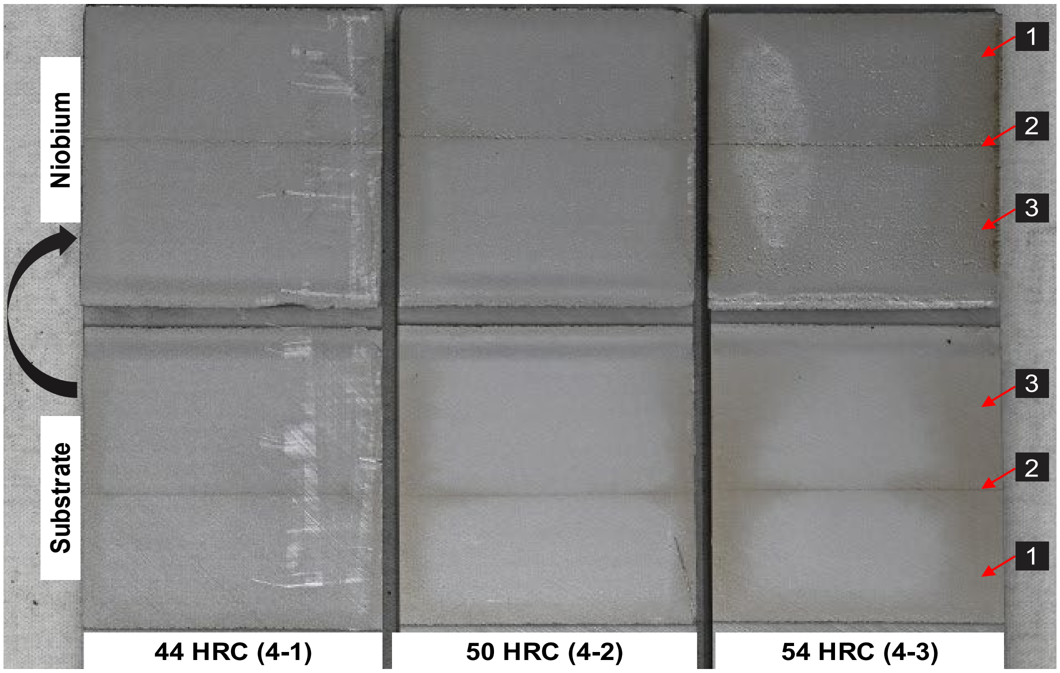

Figure 20. Delaminated surface of the Nb deposit and the 4340 steel substrate after re-applying material to the previously sprayed on area. The area indicated by (1) is the area that was not previously sprayed on, (3) was previously sprayed on, and (2) indicates the transition between each area.

Pit defects were less apparent on the coatings prepared with 4340 substrates compared with the 440C substrates. Coatings applied to the 54 HRC 4340 substrate did not exhibit premature release like the 54 HRC 440C during the first coating application, even with a similar initial Ra. Substrate Ra was measured with a stylus profilometer to keep track of the substrate state before and after each spray and substrate preparation step. The results are displayed in Figure 21, and an explanation of the conditions described are given in Table 9.

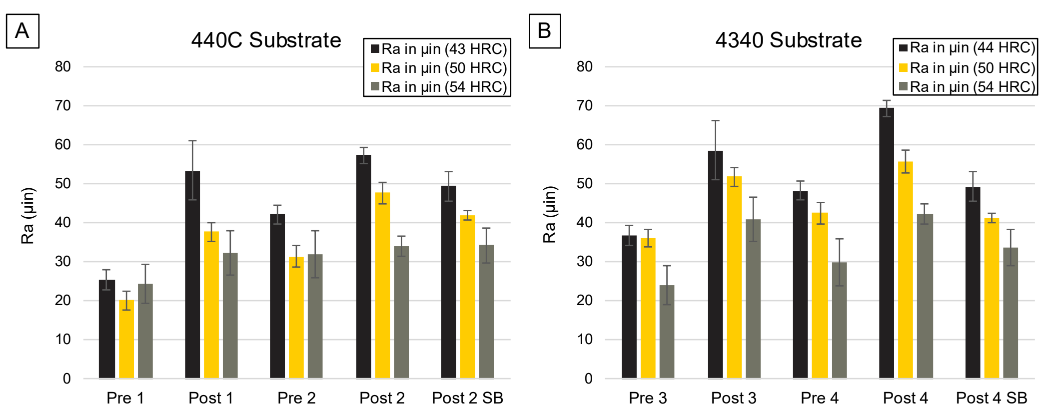

Figure 21. Surface Ra of the substrate recorded with a stylus profilometer before (Pre) and after (Post) each spray condition and after cleaning with Scotch-Brite (SB). The area of the hard sample in spray 1 and 2 measured was the portion that was not sprayed on with topcoat when the original deposit delaminated.

Table 9. Explanation of labeling in Figure 21.

Identifier	Explanation of plate condition
Pre 1/3	Plate before any spray, cleaned with Scotch-Brite (SB) and alcohol
Post 1/3	Plate after spray 1/3 release, no other surface treatment
Pre 2/4	Plate after spray 1/3, cleaned with SB and alcohol
Post 2/4	Plate after spray 2/4 release, no additional treatment
Post 2/4 SB	Plate after spray 2/4, cleaned with SB pad and alcohol

Height maps of the deposit release surfaces were constructed using a laser scanning microscope as a compliment to the photographs. The 440C and 4340 substrate coupons are displayed in Figures 22 and 23, respectively.

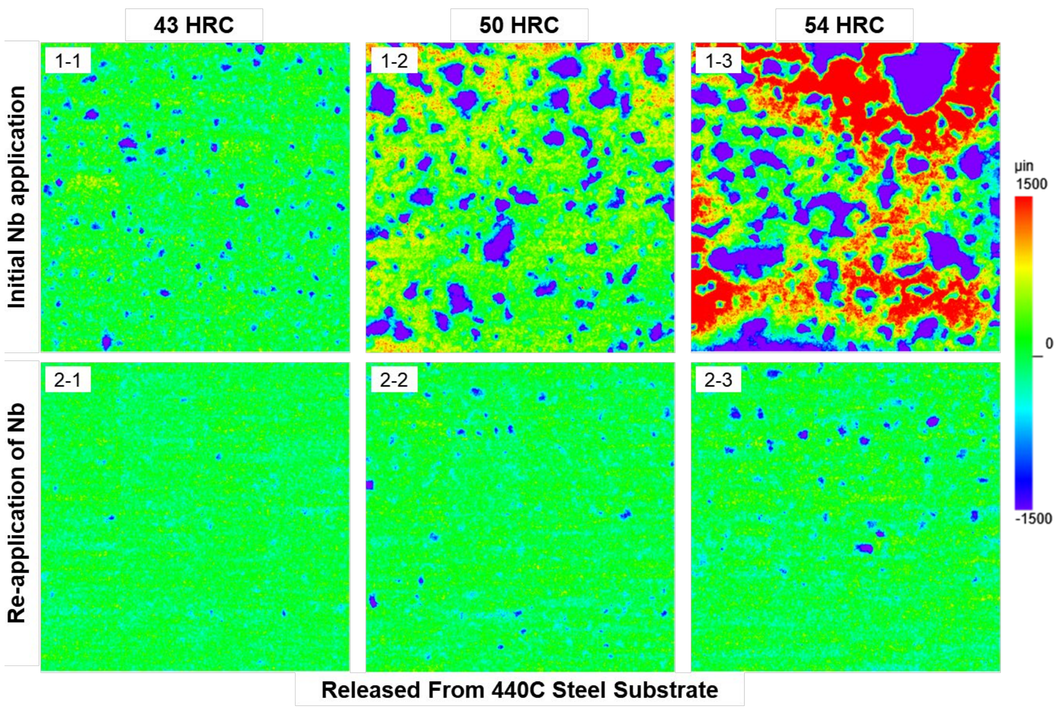

Figure 22. Height maps of the delaminated surfaces of Nb deposits sprayed onto 440C substrates of varying hardnesses. Image area is 0.295 × 0.295 inch.

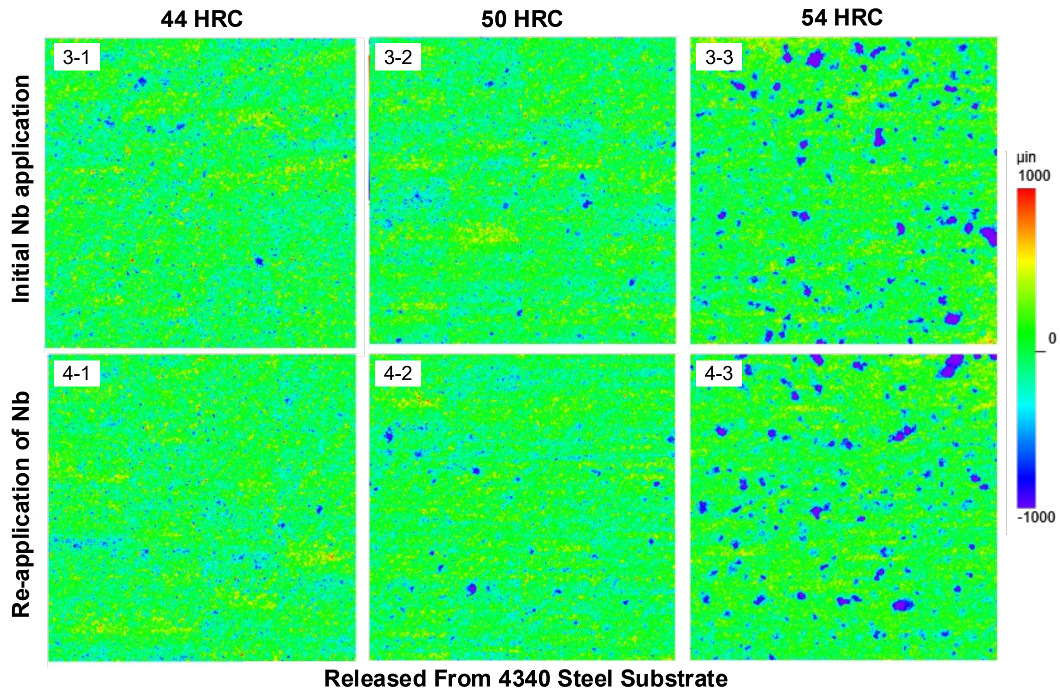

Figure 23. Height maps of the delaminated surfaces of Nb deposits sprayed onto 4340 substrates of varying hardnesses. Image area is 0.295 × 0.295 inch.

The height maps show drastic differences among each hardness, application of material (first or second), and substate type. Figure 22 depicts the drastic decrease in pit defects between the first and second application of sprayed material on the 440C substrates. Upon reapplication of material, the degree of pitting was less severe. The reduction in pitting is attributed to the substrate roughening effect that is observed from the first to the second spray application (Figure 21).

Although substrate material roughness was not explicitly controlled before coating application, it is evident that material type is a large factor in the pit defect occurrence when comparing the hardest plate of each material type. Samples 1-3 and 3-3 were prepared with similar roughness values; however, the 440C substrate produced a deposit with more defects upon the first application of material.

The Sa of Nb sprayed on the 440C substrates is highly dependent on whether the substrate was sprayed on previously and is reflected in the Sa measurements in Figure 24. The Sa of the released deposit surface varies drastically as the 400C substrate hardness is changed in the first application of material. Upon the subsequent application, however, all three materials result in a deposit with similar degrees of pit defects. In contrast, the 4340 steel substrates showed similar levels of pit defects and area roughness between sprays and between substrates, although the hardest substrate resulted in the highest degree of pit defects.

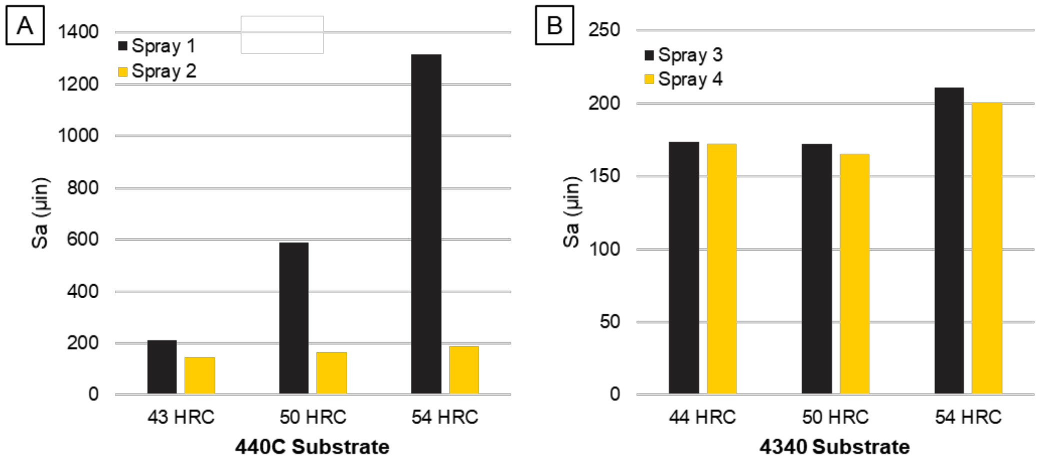

Figure 24. Sa values from the height maps generated of the deposits released from 440C substrates (A) and 4340 substrates (B).

4. Conclusions and Recommendations

The key observations from this work are summarized as follows:

1) Incorporation of preheating instrumentation in hard steel mandrels for leading edge production will reduce the severity and quantity of pit defects on part release surfaces.
2) Adhesion strength of the coatings increases with substrate temperature and decreases with substrate hardness. The presence of pit defects appears to be a function of temperature and hardness and is also strongly influenced by substrate surface roughness.
3) A hardness of 50 HRC is recommended for reducing the likelihood of premature delamination in use. A harder substrate would be viable if the appropriate surface condition is maintained between each reuse of the substrate. Harder substrates like the 54 HRC can be preheated to reduce pit defects, but it is challenging to eliminate defects on these substrates unless a rough surface finish exists, as demonstrated with a 57 HRC piece of 4340 in the grit-blasting experiment. This may be a viable means to achieve reusable tooling with high hardness materials resistant to damage during the release process.
4) The ease of deposit release from the substrate was more difficult on the softest substrate within a set of experiments.

For optimizing the release process, a series of controlled experiments is proposed to evaluate the aging properties of substrates and to further explore the relative ease of coatings release from the mandrel. Furthermore, the experiments would reveal if grit blasting can be used as a pretreatment step to achieve a roughness value on the as-manufactured mandrel close to the value obtained over time through repeat use. To explore these concepts, substrates of a given hardness should be prepared with surface grinding and grit blasting to achieve different degrees of roughness. A selection of hardness values and material types may be used, but high hardness 4340 is recommended because harder materials will be more resistant to damage from tooling used in the release process. In addition, the 4340 demonstrated more consistent performance between spray applications. The substrates should be sprayed and released multiple times until substrate roughness values plateau from one spray to the next. Moreover, the degree of surface pitting should be evaluated in the released deposit. Bond bar (ASTM C633)[3] testing or the dolly adhesion testing[2] used herein may be incorporated to evaluate how adhesion strength changes between each condition. Variables such as bond coat condition and surface preheat temperature may be investigated as needed; however, the LVBC and a 100 °C surface temperature are recommended as starting points.

5. References

1. Nault I, Nicholas M. Cold spray additive manufacturing of leading edges robotics requirements. DEVCOM Army Research Laboratory (US); 2023 Sep. Report No.: ARL-SR-0481.

2. ASTM D4541. Standard test method for pull-off strength of coatings using portable adhesion testers. ASTM International; 2022.

3. ASTM C633. Standard test method for adhesion or cohesion strength of thermal spray coatings. ASTM International; 2021.

Appendix. Release Surface Characteristics of Coatings

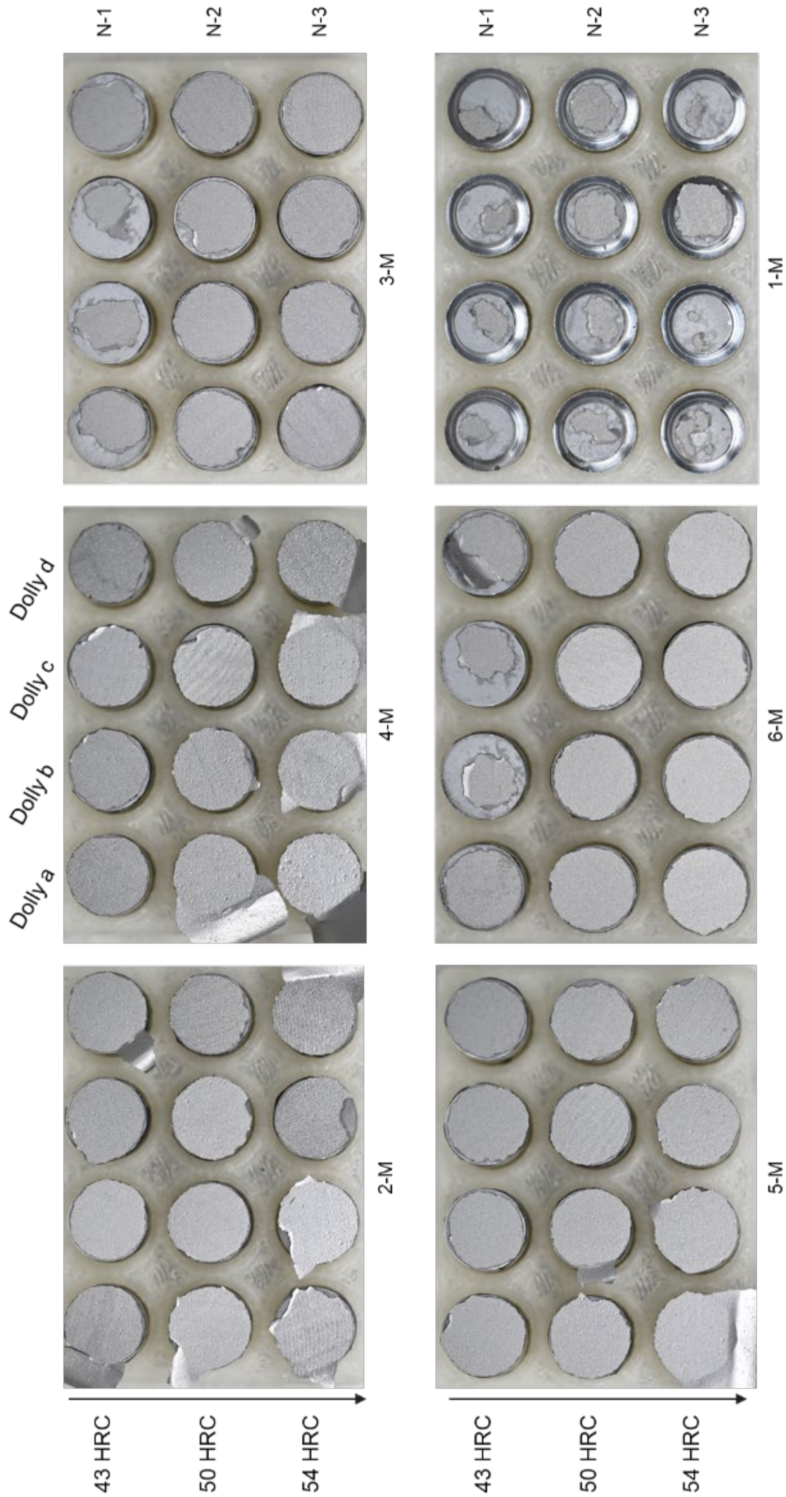

Figure A-1. Photographs of delaminated surfaces for each dolly obtained by adhesion testing. HRC = hardness on the Rockwell C scale.

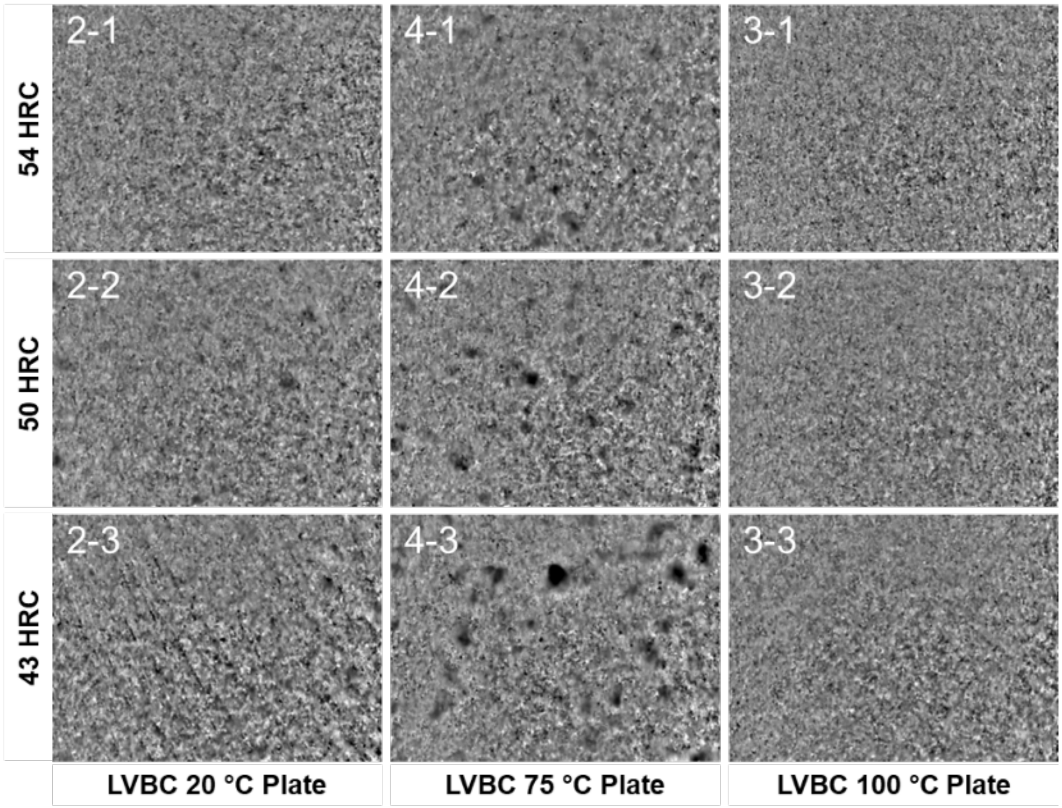

Figure A-2. Qualitative surface height for each condition from a representative area of the delaminated deposit after dolly adhesion testing. High spots are represented by lighter areas, whereas dark areas represent low spots on the image. Low-velocity bond coat (LVBC) spray condition height maps.

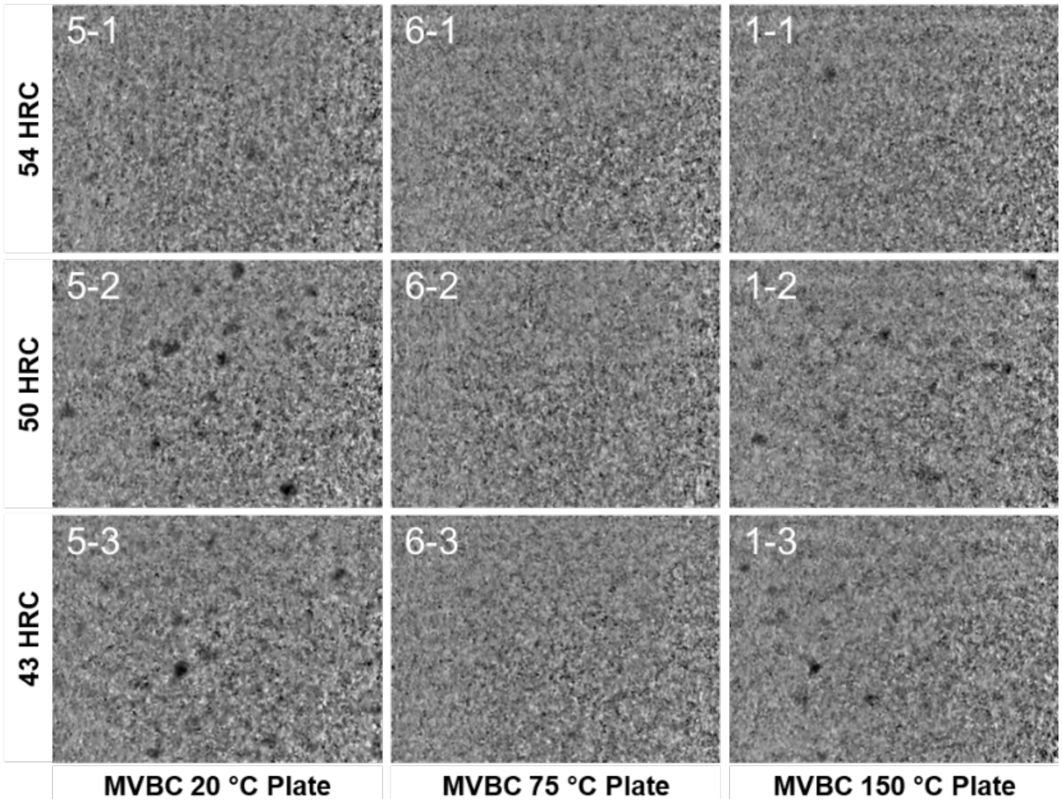

Figure A-3. Qualitative surface height for each condition from a representative area of the delaminated deposit after dolly adhesion testing. High spots are represented by lighter areas, whereas dark areas represent low spots on the image. Medium-velocity bond coat (MVBC) spray condition height maps.

List of Symbols, Abbreviations, and Acronyms

He	helium
HRC	hardness on the Rockwell C scale
LVBC	low-velocity bond coat
MVBC	medium-velocity bond coat
Nb	niobium
P_{spray}	spray system pressure
Ra	line roughness measurement
Sa	area surface roughness
SLM	standard liters per minute (gas flow rate)
TcC	temperature control
T_{plate}	plate temperature
T_{spray}	spray system temperature
µin	microinch

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