

NASA/CR—2007-214706



# Development of Oxidation Resistant Coatings on GRCop-84 Substrates by Cold Spray Process

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Prepared under Purchase Order NNC05QA42P

National Aeronautics and  
Space Administration

Glenn Research Center  
Cleveland, Ohio 44135

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May 2007

This work was sponsored by the Fundamental Aeronautics Program  
at the NASA Glenn Research Center.

*Level of Review:* This material has been technically reviewed by NASA expert reviewer(s).

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# Development of Oxidation Resistant Coatings on GRCop-84 Substrates by Cold Spray Process

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## 1. Introduction

This report is a section of the final report on the GRCop-84 task of the Constellation Program and incorporates the results obtained between October 2000 and September 2005 when the program ended.

A new copper alloy developed at NASA Glenn Research Center (GRC), containing 8%-Cr and 4%-Nb (GRCop-84) has very attractive high temperature creep and fatigue capabilities for rocket engine liners and other high temperature applications such as Concast molds, nozzles, etc. For instance, expander cycle upper stage engines that utilize a copper liner with enhanced heat transfer features could produce significant thrust to enable much higher mass payloads to space. However, experiments have shown that the capability of GRCop-84 could be extended to even higher temperatures if it can be protected from degradation due to high temperature oxidation. In order to exploit the advantageous of GRCop-84's unique characteristics in any high temperature oxidizing applications, one needs to develop oxidation resistant coatings on the hot surface by a surface coating technology such as a thermal spray process.

Thermal sprayed coatings have achieved success as both wear resistant and oxidation resistant surfaces, and numerous jet engine components are routinely used with reliable environmental barrier coatings. However, thermal spray processes have not been fully developed for copper alloys used in rocket combustion chambers, and may in fact have some inherent disadvantages stemming from the relatively low melting point and high conductivity of copper. This may result in higher than necessary porosity, oxides, and other coating defects. Therefore, this task was initiated to explore the potential advantages of a new coating deposition process known as cold spray.

The cold spray process uses high velocity rather than high temperature to produce coatings, and thereby avoids or minimizes many deleterious high temperature reactions, which are characteristics of typical thermal sprayed coatings. Typical advantages of cold sprayed coatings include compressive rather than tensile stresses, wrought-like microstructure, near theoretical density coatings, coatings free from oxides and other inclusions, etc. Moreover, the footprint of the cold spray beam is very narrow yielding a high-density particle beam, which results in high growth rate of coating thickness with better control over the shape of the coating, without masking requirements. The need for pre-spray surface roughening has been minimized or completely avoided with many substrates during the cold spray process. This results in a smoother and higher performance coating-substrate interface.

These cold spray advantages can be gainfully exploited for producing engineered bulk forms and coatings for aerospace applications such as high temperature corrosion resistant surfaces. Preliminary studies have shown that it is possible to produce cold spray coatings with very high corrosion resistant characteristics. Using this knowledge base, an experimental program was carried out to develop the cold spray technology to produce high temperature oxidation resistant surfaces for aerospace engine applications.

## 2. Cold Spray Process

When particle-laden gas jet impinges on a solid surface three different phenomena can occur depending on the particle velocity ( $V_p$ ). When  $V_p$  is low, the particles bounce off the surface. When  $V_p$  reaches moderate values, solid particle erosion takes place. When  $V_p$  exceeds a critical velocity (characteristics of the powder material), particles plastically deform and adhere to the substrate and one

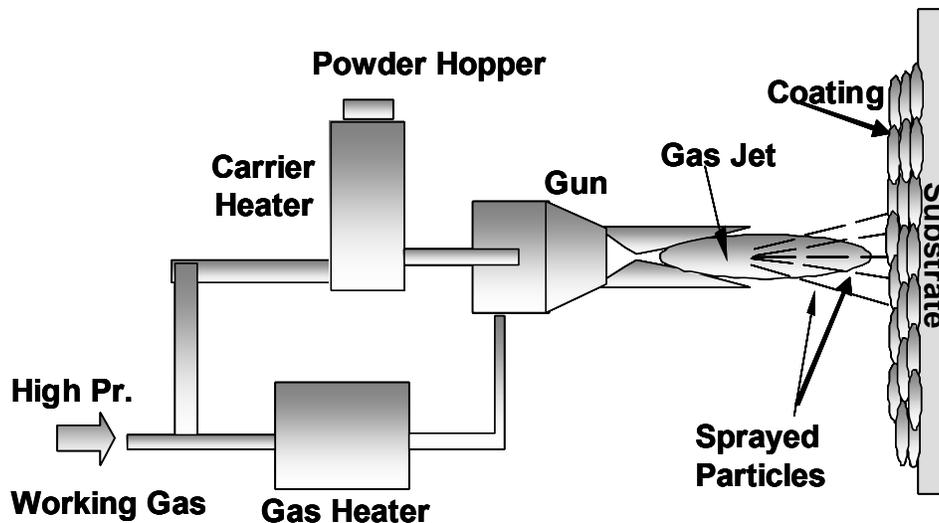


Figure 1.—Schematic of cold spray process.

another forming a dense adherent coating. Russian scientists have developed this phenomenon into a high deposition spray process.

Figure 1 shows the schematic of the Cold Spray system. The heart of the system is the spray gun, which contains a converging-diverging nozzle to impart supersonic velocity to the gas and a mixing chamber where the powder particles are injected into the high-pressure gas stream at exact location. Diagnostic ports to measure and control the gas pressure and temperature are incorporated in the mixing chamber. High-pressure (up to 30-bar/500 psi) gas such as nitrogen, helium and their blends are used as the working gas. In order to compensate for the cooling associated with the rapid expansion at the nozzle, an electric heater is used to preheat the working gas to about 500 to 800 K (450 to 1000 °F). A high-pressure hopper is used to feed the powder. Powders used are typically in the range of 1 to 40  $\mu\text{m}$  in diameter. Conventional job handling systems such as the X-Y manipulator and a lathe are used to scan the spray beam over the substrate surface.

### 3. Experimental Program

Four different materials, including three proprietary CuCrAl compositions of NASA GRC and a NiCrAlY material were used as spray feedstock. Both single layer and duplex coating systems were produced. GRCop-84 plates and disks were used as substrates. Necessary substrate holders and spray setups were designed and fabricated. Microstructure study was used to optimize the spray process for producing good quality coatings of each of the four powders. Plate specimens and disks were spray coated with about 0.010 in. thick coating for bond test, flame test and diffusion studies. GRCop-84 buttons (0.5 in. diam. by 0.0080 in. thick) were sprayed with CuCrAl coatings on all sides including the edges for investigation of oxidation resistance characteristics. Ultrathick coatings were produced over expendable mandrels and subsequently removed for producing mechanical test specimens, thermal property measurement specimens, etc. Following sections give details of various aspects of the program activities.

#### 3.1. Coating Systems

NiCrAlY is a standard material for producing oxidation resistant coatings. This material was selected as one of the test materials. Various compositions of NiCrAlY and CoCrAlY are available for producing thermal spray coatings for different applications. For this program, a standard NiCrAlY (Praxair Ni 346-

5) powder was purchased from Praxair and used as-received for spray producing NiCrAlY coatings and duplex coatings. Different Cu-Cr-Al alloys have been invented at NASA GRC which shows excellent high temperature oxidation resistance. Three CuCrAl compositions with different chemical compositions were studied. Thus, a total of four different materials, given below, were studied

- Ni-17 wt% Cr-6 wt% Al-0.5 wt% Y
- Cu-8 wt% Cr-1 wt% Al
- Cu-8 wt% Cr-5 wt% Al
- Cu-23 wt% Cr-5 wt% Al

NASA GRC supplied 50 lb each of the four materials for spray experiments and producing deliverables.

### 3.2. Substrates

OFHC copper plates (6 by 6 by 0.125 in.) were used as substrates for spray optimization experiments. Copper coupons of dimensions 2 by 3 by 0.125 in. were used for producing coatings for microstructural investigations.

In order to produce free standing cylinders of CuCrAl material for mechanical test specimen fabrication, expendable 4 in. i.d. by 0.25 in. wall by 4 in. long aluminum tubes were used as mandrels. The aluminum tubes were removed by mechanical and chemical means to produce 4 in. diam. by 4 in. long by 0.330 to 0.380 in. thick CuCrAl pipes.

For producing high temperature test specimens and oxidation specimens, coatings were produced over NASA supplied GRCop-84 substrates. NASA GRC supplied 36 GRCop-84 disks, nominally 0.5 in. diam. by 0.160 in. thick and 8 GRCop-84 plates, nominally 6 by 8 by 0.160 in. thick.

### 3.3. Cold Spray Development Study

Initial spray experiments were conducted with NiCrAlY and Cu8Cr5Al powders. It was observed that both powders lead to nozzle fouling issues, namely blockage of nozzle and gas flow due to deposition of powder particles in the nozzle walls. Earlier experiments with CuCrAl powder have shown that spray operation with helium and at high temperature produce excellent coatings. When experiments were tried to repeat the spray operation with same parameters, nozzle fouling occurred within about 2 min. When standard steel nozzles are used, it is possible to spray both powders only for a short time (<2 min) when the system operating parameters are optimized to produce high quality coatings. Since the compositions are similar, the other two powders were expected to have similar characteristics. It is possible to have long duration spray runs with reduced parameters and produce coatings with nitrogen or lower temperature. However, the coating characteristics are poor (high porosity and low strength) with very low deposition efficiency. Such low-efficiency operation is not suitable for the present work.

Next, spray experiments were carried out by switching from a metal nozzle to an HM nozzle which was developed for spraying nozzle-fouling materials such as aluminum. However, since the powders have high chromium and resultant hardness as compared to aluminum, the nozzle throat was eroded quickly. In the cold spray process, the particle velocity is dependant on the inlet pressure and expanded throat will lead to lowering of the gun pressure. The spray system operates under feedback controlled pressure regulation mode. This allows higher gas flow to compensate for the throat erosion. Thus a HM nozzle can be used to run the spray experiments for up to 20 to 30 min. Since the mechanical test cylinder fabrication requires much longer duration spray, it was decided to achieve long duration spray before any microstructure specimens were produced.

CGT Technologies, Germany, had developed a new tungsten carbide nozzle. They have tested this nozzle and found that many hard materials can be sprayed without fouling with this nozzle. These nozzles are pressed, sintered, and machined to designed dimensions. The internal bores are diamond honed to

finish the nozzles. Two nozzles were purchased from CGT, and these nozzles were found to perform satisfactorily. A series of spray experiments, given below were carried out with each powder.

### 3.4. Cold Spray Process Optimization

We have already carried out preliminary spray experiments with typical NiCrAlY and CuCrAl materials. These studies have shown that cold spray process parameters have strong effect on the process characteristics, as well as on the properties of the coatings. Results of these studies were used as the basis, and screening experiments were carried out to establish the effect of process parameters on the properties of the coatings. Important spray process parameters include

- Temperature of the working gas
- Gas composition
- Gas pressure
- Powder feed rate
- Carrier gas feed rate

These parameters were varied, and with each set of spray parameters, about 20 mil thick coatings were produced and subjected to microstructural investigations.

### 3.5. Microstructural Characterization of the Coatings

Microstructural investigations of the coatings were carried out to establish the variation of coating characteristics with process parameters, and use these results to optimize the process parameters. Coatings were characterized in terms of density, porosity, coating-substrate interface characteristics and other microstructural features. Samples were cut, vacuum impregnated with dyed epoxy, mounted, ground and polished, and investigated with optical microscope. Microstructural features of the samples were recorded using digital microscopy.

Figure 2 shows typical microstructure of different NiCrAlY specimens, produced with various spray parameters. As can be seen, in general a dense layer of thick coating is formed. There is a slight variation in the coating characteristics with spray parameters. However, these variations are not significant.

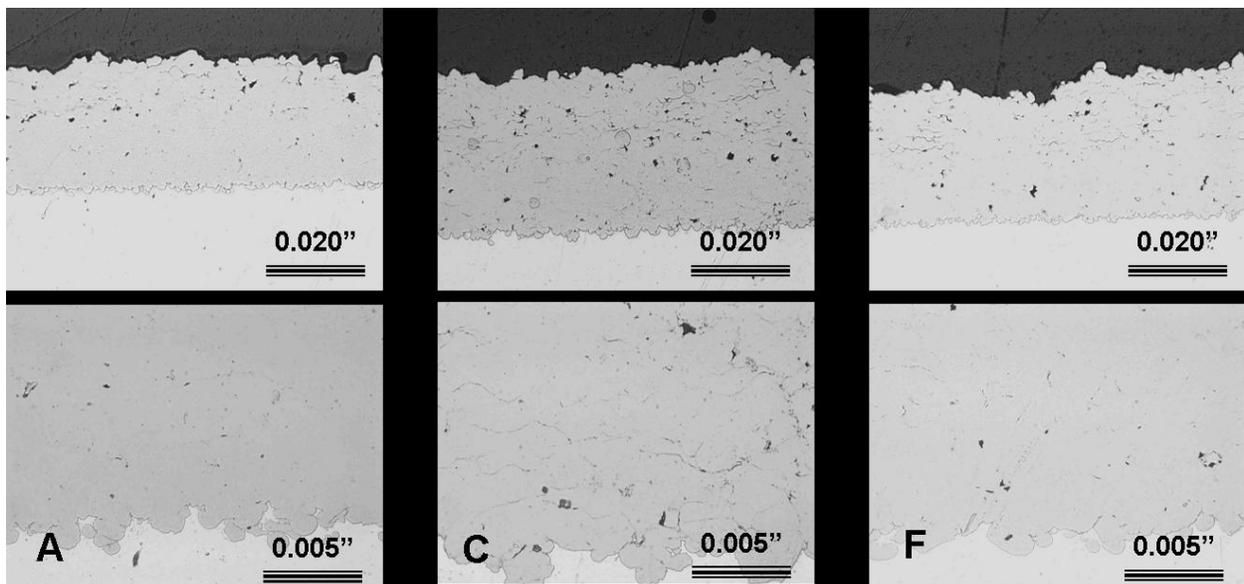


Figure 2.—Variation of coating microstructure with process parameters.

Figure 3 gives the microstructure of a typical specimen. This shows that a dense layer is formed at the bottom, while some porosity is visible on the top. It should be noted that these specimens have two passes sprayed and it is well known in cold spray process that the second layer shot peens and densifies the first layer. This shows that the deliverables should be sprayed with multiple passes so that bulk of the coating will have dense structure.

Similar spray optimization studies were carried out with each of the four powders. Typical coatings were produced over copper substrates with different sets of spray parameters. Representative coatings were subjected to microstructure studies and based on these results, process parameters were optimized. Typical microstructure of optimized coatings is given in figure 4. As can be seen, dense layers are obtained with excellent bonding to copper substrate. Since GRCop-84 has similar characteristics, it is believed that the bond strength of Cu8Cr5Al over GRCop-84 substrate will be similarly high.

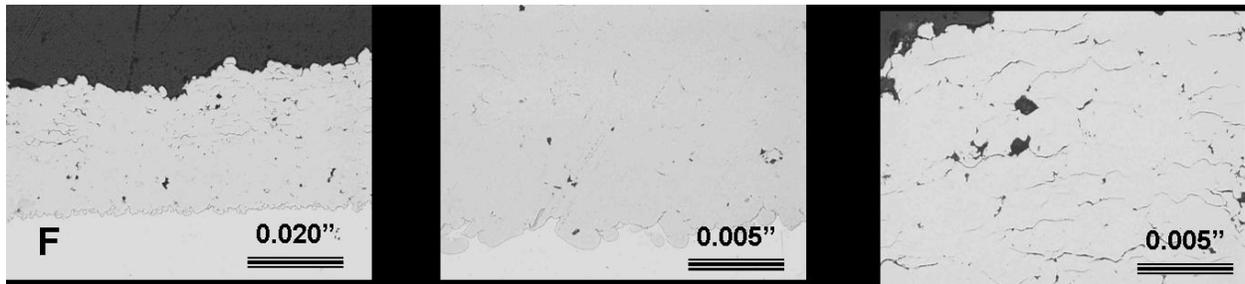


Figure 3.—Variation of microstructure across the coating thickness.

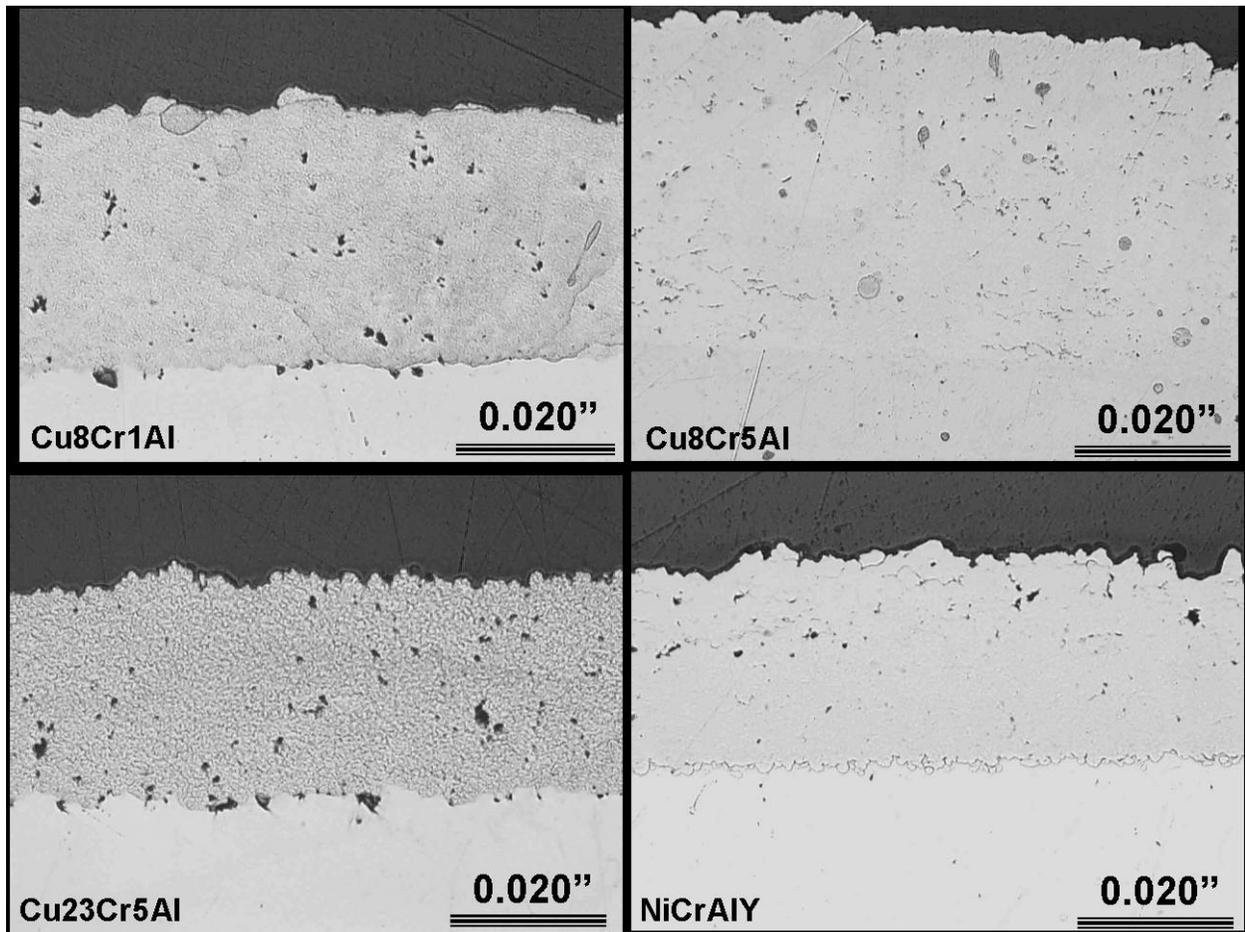


Figure 4.—Microstructure of optimized coatings.

### 3.6. Cold Spray Deliverables

Tooling, required for producing the deliverables was designed and built by ASB. Once the process was optimized and spray techniques evolved, preliminary spray experiments were carried out to fine tune the parameters and subsequently produce deliverables.

#### 3.6.1. Tooling and Preliminary Experiments

For spraying the 0.5 in. diameter disk substrates for oxidation studies, a turn-table with appropriate specimen holders was designed and built to ensure that coatings with uniform thickness can be produced over entire surfaces of the disk, as well as maximum coverage of the disk edges. Figure 5 gives the oxidation specimens spray set up. Initial experiments were carried out with OFHC copper powder and process parameters such as the rotation speed, scan rate, powder feed rate, etc., were optimized.

NASA GRC supplied a sufficient numbers of dummy and deliverable GRCop-84 substrates (both oxidation buttons and plate specimens) for both process validation studies and for producing deliverables. Coated samples on dummy GRCop-84 substrates were produced to study the substrate effects with each powder. Initial experiments were carried out to obtain good quality coatings with acceptable bonding to GRCop-84 substrate material. Optimized parameters were used to operate the spray system, and the experiments concentrated on surface preparation techniques, coating deposition rate, scan parameters, etc, to obtain uniform coating and maximum bond strength with minimum surface roughness. Coating thickness of these specimens was set as  $0.012 \pm 0.002$  in. Once the entire procedure, parameters and technique have been established, additional specimens were spray produced as deliverables.

#### 3.6.2. CuCrAl Adhesion Tests, Torch Test, and Diffusion Studies Specimens

First, Cu<sub>8</sub>Cr<sub>5</sub>Al powder was loaded and the spray system was operated at optimized parameters for spraying the Cu<sub>8</sub>Cr<sub>5</sub>Al powder. Dummy copper substrates were installed and spray experiments were carried out, varying the powder feed rate, gun scan rate and step size to obtain the coating thickness per pass to the required value of 0.010 to 0.020 in. Then, the GRCop-84 substrate spray was taken up. Figure 6 shows the processing of plate specimens. Two 6 in. wide by 8 in. long GRCop-84 substrates were cleaned with alcohol, grit blasted at low pressure to clean the surface but avoid grit inclusion, and mounted onto the spray set up. Operating the spray system with preset values, two passes were sprayed to obtain 14 to 18 mils coating on the entire surface of the plates.

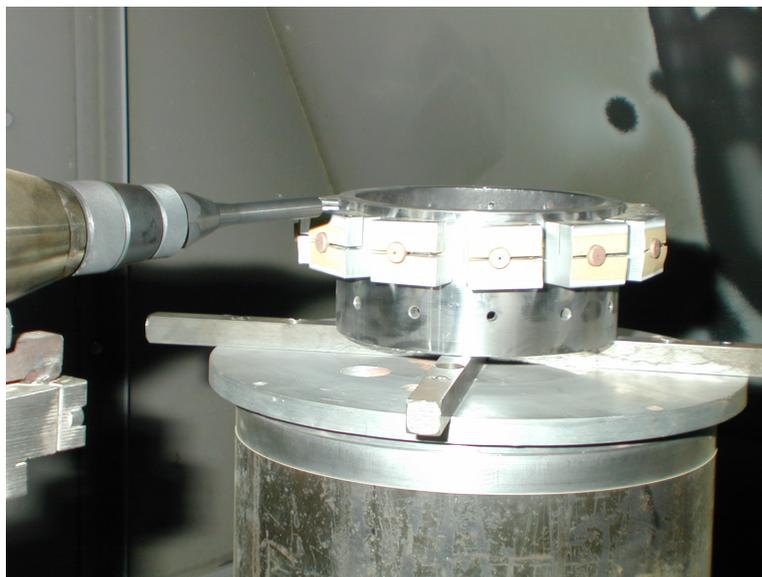


Figure 5.—Oxidation button spray set-up.



Figure 6.—Cold spray processing of plate specimens.

Then, similar experiments were carried out with Cu<sub>23</sub>Cr<sub>5</sub>Al powder and another set of two 6 by 8 in. GRCop-84 plates were sprayed with 0.016±0.002 in. coating of Cu<sub>23</sub>Cr<sub>5</sub>Al powder.

Finally, similar experiments were carried out with Cu<sub>8</sub>Cr<sub>1</sub>Al powder and another set of two 6 by 8 in. GRCop-84 plates were sprayed with 0.006 to 0.007 in. coating of Cu<sub>8</sub>Cr<sub>1</sub>Al powder to complete the work.

### 3.6.3 CuCrAl Oxidation Specimens

Initially, a set of dummy copper disks were installed on special substrate holders and mounted onto the turntable. Rotation speed of the turntable, powder feed rate and scan step size were controlled to produce about 0.002 in. thick coating per pass.

A set of 12 GRCop-84 disks of 0.5 in. diameter were installed on the turn-table and sprayed 7 passes with a 40 rpm table rotational speed. These parameters yielded 0.017 to 0.018 in. thick Cu<sub>8</sub>Cr<sub>5</sub>Al coating on the face of the disks. Then, these disks were removed from the holder, turned around, remounted and sprayed with 0.017 to 0.018 in. thick Cu<sub>8</sub>Cr<sub>5</sub>Al coating on the other side. However, it was observed that the disks have sharp edges and this resulted in coating failure in some specimens. Coating had flaked off partially or completely in three specimens leaving only 9 good specimens with Cu<sub>8</sub>Cr<sub>5</sub>Al coating.

It was believed that the specimen clamping and spray angle could be set so that the disk edges will also get sprayed by the overspray. However, it was observed that the edges did not receive any coating. Since the oxidation specimens require the entire coupons to be encapsulated by the coating, another spray step, shown in figure 7, was introduced to produce coating on the edges of the specimens. A quick scan of the spray pattern over the edge yielded a well bonded coating of about 10 mil thick coating.

Then, similar spray operation was taken up with Cu<sub>23</sub>Cr<sub>5</sub>Al powder. The edges of 12 oxidation specimens were first chamfered to avoid bond-failure. The samples were cleaned, grit blasted and sprayed following exactly similar procedure. Measurements showed that these specimens have 13 to 15, 15 to 16 and 10 to 12 mil thick coatings on the two sides and edge of the disks, respectively.

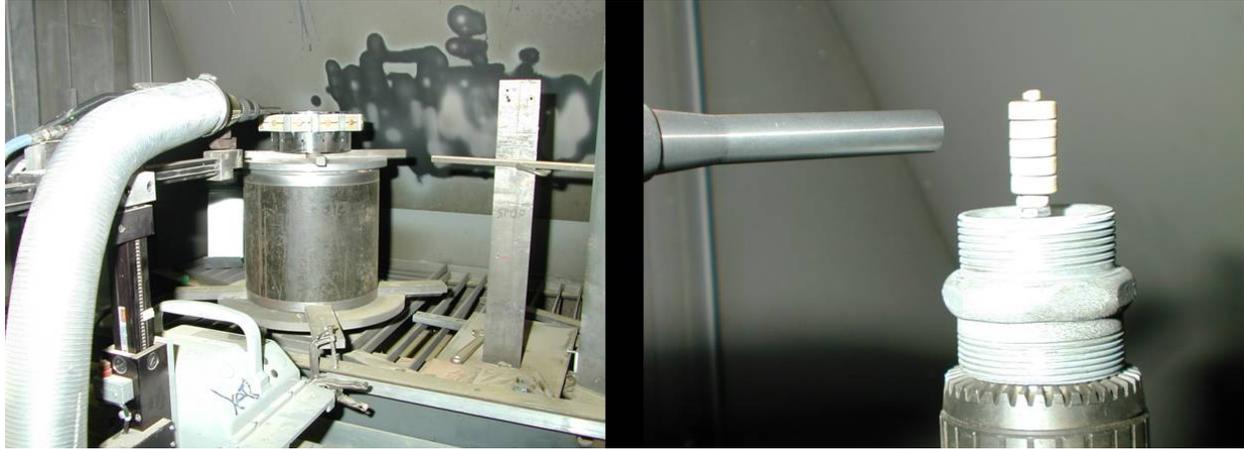


Figure 7.—Cold spray processing of the oxidation specimens.  
Left image shows spraying the faces and right shows spraying the edge.

### 3.6.4 CuCrAl Mechanical and Physical Properties Specimens

Monolithic 4 in. diam. by 4 in. long by 0.300 in. thick CuCrAl pipes are required for producing specimens for measuring the mechanical and physical properties of the CuCrAl material. Such bulk forms can be produced by first spraying over a expendable mandrel, machining the coating to required dimensions and then removing the mandrel by mechanical and/or chemical methods. We have already produced such monolithic forms of different materials such as GRCop-84, discontinuously reinforced aluminum composites, etc., by similar procedure and this experience was used for producing the CuCrAl tubes.

Aluminum tubular mandrels (4 in. diam. by 4.25 in. long by 0.25 in. wall) were used as the substrate, and optimized parameters were used to operate the spray system. The powder feed rate, scan rate and the rotational speed of the turntable were adjusted to have about 10 to 15 mils per pass. In total, three CuCrAl tubes were produced.

First the Cu<sub>8</sub>Cr<sub>5</sub>Al powder was loaded and sprayed for a total of 28 passes to get a coating thickness of about 350 mils on the first aluminum tube. Next the Cu<sub>23</sub>Cr<sub>5</sub>Al powder was loaded and sprayed 26 passes onto a second aluminum tube to get 325 mils coating thickness.

After completion of the spray operation, first the Cu<sub>8</sub>Cr<sub>5</sub>Al specimen was mounted onto the CNC machine and the aluminum tube was machined off. Unfortunately, the uneven surfaces of the sprayed specimen resulted in improper mounting and the coating developed a few cracks which were visible once the aluminum was removed. Efforts to machine out the cracked region (assuming that the cracks are only on the surface) did not succeed as the cracks were rather deep.

Hence, this specimen was abandoned and another Cu<sub>8</sub>Cr<sub>5</sub>Al specimen was produced using the same parameters. In total, 27 passes were sprayed to produce about 350 mils per side coating thickness on a third aluminum tube.

An aluminum mount was designed and built to hold the specimens for machining. This mount held the specimens compressively all around the specimens. With this mount and a reduced machining rate for removing the aluminum tube, the substrate tube could be machined off the second and third specimens.

The following specimens were delivered to NASA GRC as deliverables

- Cu<sub>8</sub>Cr<sub>5</sub>Al tube with 4 in. i.d. and 4.25 in. long with about 0.350 in. wall thickness.
- Cu<sub>23</sub>Cr<sub>5</sub>Al tube with 4 in. i.d. and 4.25 in. long with about 0.350 in. wall thickness.
- Apart from the deliverables, the first Cu<sub>8</sub>Cr<sub>5</sub>Al tube with the cracks was also delivered to NASA GRC. Its dimensions were 4 in. i.d. and 4.25 in. long with about 0.150 in. wall thickness.

### ***3.6.5 Duplex NiCrAlY-CuCrAl Adhesion Tests, Torch Test, and Diffusion Studies Specimens***

NASA required Cu<sub>8</sub>Cr<sub>1</sub>Al-NiCrAlY duplex coatings for studying their oxidation resistance characteristics and comparing them with CuCrAl coatings. This required a thin (0.004±0.001 in.) bond coat of CuCrAl coating followed by about 0.008±0.001 in. thick NiCrAlY coating to be produced over GRCo-84 substrate plates and buttons. The total coating thickness was to be the same in all the three sets of specimens.

Initially, two test 6 in. wide by 8 in. long GRCo-84 plates were mounted and sprayed with optimized spray parameters for spraying Cu<sub>8</sub>Cr<sub>1</sub>Al coatings. During the spray operation, the powder feed rate and scan rates were adjusted to yield a thin (~0.005 in.) coating. This produced a non-uniform coating thickness with about 0.009 in. thick CuCrAl coating over the most of the region. Then, two 6 in. wide by 8 in. long GRCo-84 substrates were mounted and sprayed with one pass to obtain about 0.005 in. coating of Cu<sub>8</sub>Cr<sub>1</sub>Al powder.

The hopper was changed from Cu<sub>8</sub>Cr<sub>1</sub>Al to NiCrAlY and spray parameters changed to produce optimized NiCrAlY coatings. The first set of plates were installed again and sprayed one pass to obtain about 10 to 12 mils thick NiCrAlY coating. The second set of plates were installed and about 0.009 to 0.010 in. thick NiCrAlY coating was produced over the Cu<sub>8</sub>Cr<sub>1</sub>Al coatings. These four duplex coated plates were delivered to NASA GRC.

### ***3.6.6 Duplex CuCrAl-NiCrAlY Oxidation Specimens***

Initially, dummy copper buttons were installed in the turntable and sprayed with Cu<sub>8</sub>Cr<sub>1</sub>Al powder. Optimized parameters were used, and the turn table speed, scan rate and powder feed rate were adjusted to achieve coating thickness of about 0.004 in. Then a set of 12 GRCo-84 disks of 0.5 in. diameter was edge chamfered, cleaned and blasted and installed on the turn-table and sprayed with two passes to get about 0.005 in. Cu<sub>8</sub>Cr<sub>1</sub>Al coating on the entire surface. These specimens were turned and sprayed two passes to get about 0.006 in. thick coating.

Next, the powder in the hopper was changed from Cu<sub>8</sub>Cr<sub>1</sub>Al to NiCrAlY and spray parameters were changed to produce optimized NiCrAlY coatings. The samples were sprayed 8 passes each on both sides to obtain 0.010 in. thick NiCrAlY coating over the CuCrAl coating. The total thickness of the duplex coating is 0.014±0.002 in.

Next, the edge spray setup was installed and a single scan of the spray pattern with CuCrAl powder and two scans with NiCrAlY powder gave the required duplex coating on the edges as well, completely encapsulating the specimens.

## **IV. Concluding Remarks**

The cold spray process yields high purity metal alloy coatings with potentially superior characteristics. In this program, CuCrAl and NiCrAlY coatings were produced over GRCo-84 substrates by cold spray process for oxidation protection. Initially, process parameters were optimized using microstructural study as the metering. Then, a number of plates and disk specimens were produced with specific coating characteristics for subsequent studies at NASA GRC.

**REPORT DOCUMENTATION PAGE**

*Form Approved  
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<b>1. REPORT DATE (DD-MM-YYYY)</b> 02-05-2007		<b>2. REPORT TYPE</b> Final Contractor Report		<b>3. DATES COVERED (From - To)</b>	
<b>4. TITLE AND SUBTITLE</b> Development of Oxidation Resistant Coatings on GRCop-84 Substrates by Cold Spray Process				<b>5a. CONTRACT NUMBER</b>	
				<b>5b. GRANT NUMBER</b>	
				<b>5c. PROGRAM ELEMENT NUMBER</b>	
<b>6. AUTHOR(S)</b> Karthikeyan, J.				<b>5d. PROJECT NUMBER</b> NNC05QA42P	
				<b>5e. TASK NUMBER</b>	
				<b>5f. WORK UNIT NUMBER</b> WBS 599489.02.07.03.06	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> ASB Industries, Inc. 1031 Lambert St. Barberton, Ohio 44203				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> E-15931	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> National Aeronautics and Space Administration Washington, DC 20546-0001				<b>10. SPONSORING/MONITORS ACRONYM(S)</b> NASA	
				<b>11. SPONSORING/MONITORING REPORT NUMBER</b> NASA/CR-2007-214706	
<b>12. DISTRIBUTION/AVAILABILITY STATEMENT</b> Unclassified-Unlimited Subject Category: 26 Available electronically at <a href="http://gltrs.grc.nasa.gov">http://gltrs.grc.nasa.gov</a> This publication is available from the NASA Center for AeroSpace Information, 301-621-0390					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> GRCop-84, a Cu-CR-Nb alloy, has been developed for rocket engine liner applications. For maximum life additional oxidation protection is required to prevent blanching. NiCrAlY was identified as a suitable coating, and efforts were initiated to develop suitable coating techniques. Cold spray is one technique under consideration. Efforts at ASB Industries to produce dense, adherent coatings are detailed. The work culminated in the production of samples for testing at NASA Glenn Research Center.					
<b>15. SUBJECT TERMS</b> Copper; Coatings; Thermal barrier					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b> 15	<b>19a. NAME OF RESPONSIBLE PERSON</b> David L. Ellis
<b>a. REPORT</b> U	<b>b. ABSTRACT</b> U	<b>c. THIS PAGE</b> U			<b>19b. TELEPHONE NUMBER (include area code)</b> 216-433-8736



