Metallurgical and Process Comparison of Vacuum Plasma Spray Forming on Internal and External Surfaces--A Technical Note

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Vacuum plasma spray (VPS) forming is being developed and characterized for near-net-shape fabrication of aerospace components. Applications require VPS forming of structural materials in both monolithic form (i.e., freestanding shapes) and as integral parts of complex components (e.g., a liner for a rocket engine comhustor). In these applications the material deposited on both the inside and the outside of large and small components must meet strict quality requirements. This paper discusses metallurgical and processing comparisons between depositing material on inside and outside surfaces of symmetrical shapes. Specific examples of material properties (e.g., grain structure, hardness, and tensile properties) and process parameters (e.g., standoff distance and gun design) are discussed in terms of the fabrication of large rocket engine combustion chambers.

1. Introduction

VACUUM plasma spray (VPS) forming of near-net-shape components is being developed at NASA's Marshall Space Flight Center. In the earliest applications of VPS forming, material was deposited on the outside diameter (OD) of a structure to complete the component, or around a mandrel that was subsequently removed to form a freestanding shape (Ref 1,2). Later this process was adapted to the spray forming of material on the inside surface of larger structures, provided that the inside diameter (ID) was large enough to accommodate the insertion of the plasma gun. Although this transition may seem like a simple matter of geometry, several obstacles had to be overcome to provide an acceptable and repeatable procedure for the VPS of internal surfaces.

This paper reports on the development of VPS forming of both ID and OD spray orientations. Metallurgical and processing considerations for each spray orientation are compared. Spray forming of liquid rocket engine combustion chambers (Ref 3) provides the most direct comparison of ID and OD appli cations.

2. Background

Vacuum plasma spray forming can be defined as the deposition of a thick, $(\geq 6.4 \text{ mm})$, or 0.25 in.) coating onto a substrate. Vacuum plasma spray forming is capable of high material-deposition rates (6.8 kg/h, or 15 lb/h, for copper-base alloy) making it an attractive method for forming combustion chamber liners

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for liquid rocket engines. NARIoy-Z is a copper-base alloy (Ref 4, 5) with a nominal composition of Cu-3.0Ag-0.5Zr and high thermal conductivity $(355 W/m \cdot K; 80\%$ International Annealed Copper Standard, or IACS). It is used in the combustion chamber liner of the Space Shuttle main engine and is the baseline material for current VPS forming development.

This development work has focused on two fabrication sequences for combustion chamber liners. One sequence deposits the liner on the OD of a removable mandrel; the other spray forms the liner in the ID of a combustion chamber jacket. Consequently, a great deal of NARloy- Z^{M*} specimens have been sprayed and evaluated in each orientation. The VPS process was selected for evaluation because of all the thermal spray processes it provided deposits of the highest quality (e.g., strength, conductivity, density, bonding, etc.) and was more economical and feasible than vapor deposition and plating processes.

3. Comparison of OD and ID Processing

In this study, OD spray forming refers to deposits made on substrates such as round bars, tubes, flat plates, or contoured mandrels (Fig. 1). The most striking difference between OD and 1D spray forming is the distance from plasma torch to workpiece. That is, ID sprays are restricted by the inside diameter being sprayed. Without this restriction, the operator has the flexibility of selecting the optimum standoff distance without compromising other parameters. A standoff of 25 to 40 cm (10 to 16 in.) is typical, depending on the part size. Larger specimens usually require less standoff because they provide a large heat sink and thus are less susceptible to overheating from the plasma torch. Adequate heating is essential to ensure a recrystallized microstructure in the deposit (Ref 6). The OD spray-forming method is the more common and straightforward configuration.

The ID spray-forming process is more restrictive and thus more demanding in terms of parameter flexibility and hardware

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Fig. 1 Vacuum plasma spray forming of NARIoy-Z on the outside of a combustion chamber mandrel

Fig. 3 Electro Plasma Inc. VPS spray guns. A, EPI-08CA; B, EPI-07CA; C, EPI-03CA; D, EPI-04CA

Fig. 2 Vacuum plasma spray forming of NARIoy-Z on the inside of a 305 mm (12 in.) diam pipe mandrel

performance (Fig. 2). Placing the plasma gun inside the part imposes obvious restrictions on the torch-to-workpiece distance. Less obvious are the demands that ID spraying places on the spray hardware. The smaller working volume requires smaller plasma guns. Typically, a bulky gun is a significant limitation, especially when spray forming on contoured shapes that require multiaxis motion. Radiated heat from the substrate is also an important consideration. In ID spray forming, the plasma gun is surrounded by a very hot part—typically at 760 to 1095 $^{\circ}$ C $(1400 \text{ to } 2000 \text{ °F})$, depending on the material. The intense heat radiating from the part imposes severe limitations on the plasma gun.

To increase ID spray thickness, a plasma gun must be thermally hardened for the more demanding environment. In close consultation with the manufacturer, Electro Plasma Inc. (EPI), the existing 120 kW model EPI-03 was redesigned. All exposed plastics were removed or shielded within the torch, and water

Table 1 Parametric values for OD and ID VPS of NARIoy-Z

cooling was incorporated. The resulting model became the EPI-07CA (Fig. 3) and was used for VPS in ID sprays where the part diameter was 250 mm (10 in.) or larger (smaller-diameter pieces have been coated using the 07CA gun, but not with an orthogonal spray). To address the torch size problems associated with ID spraying, the 55 kW EPI-04CA model was also thermally hardened and became the EPI-08CA minigun (Fig. 3). The 08CA gun was used to ID spray 180 to 250 mm (7 to 10 in.) diam parts (as with the 07CA, smaller diameters can be coated, but at less than 90° impact).

Overheating limitations were not confined to the plasma guns. The Copper-base NARloy-Z powder in the feed lines (tubes) became sufficiently hot to sinter into clumps and clog in the tubes or inside the plasma gun. Water cooling was added via a coaxial jacket placed around the existing powder feed lines.

These hardware modifications, combined with parameter adjustments provide continuous ID spray duration to 8 h for NAR-

(a) (b)

Fig. 4 Microstructure of VPS NARloy-Z (HIP with heat treatment) on the inside of a 305 mm (12 in.) diam pipe. (a) Unetched. (b) Etched with ammonium persulfate solution

Fig. 5 Microstructure of VPS NARloy-Z (HIP with heat treatment) on the outside of a 127 mm (5 in.) diam pipe. (a) Unetched. (b) Etched with ammonium persulfate solution

loy-Z. Table 1 compares the parameters used for OD and ID spray forming of NARloy-Z. Note that all OD and ID sprays were conducted in an orthogonal orientation with respect to the substrate. Multiaxis motion was used to maintain torch normality.

4. Results

4.1 *Microstructure*

Microstructures of ID plasma sprayed and hot isostatically pressed (HIP'ed) NARloy-Z are shown in Fig. 4. All specimens discussed here received the following thermal treatment after spraying: HIP cycle of 105 MPa (15 ksi) pressure at 940 °C (1725 °F) for 3 h, followed by heat treatment from room temperature to 750 $^{\circ}$ C (1380 $^{\circ}$ F) over 2 h, hold for 16 h at the same temperature, 2 h ramp down to 480 $^{\circ}$ C (900 $^{\circ}$ F), hold for 4 h, ramp down to 175 $\rm{°C}$ (350 $\rm{°F}$) over 6 h, and furnace cool. The unetched microstructure (Fig. 4a) is very dense with no visible oxide. Porosity is less than 1% as measured by a Leco 2001^{TM} (Lowndes Engineering Co., Inc., Valdosta, GA) image analyzer. The etched microstructure of this sample reveals a totally recrystallized structure that is similar to wrought material, with an ASTM grain size of approximately 6 to 7 (\sim 60 µm).

Table 2 Knoop microhardness data for ID and OD VPS NARIoy-Z (HIP and heat treated)

Using the same powder chemistry and plasma gun, specimens were sprayed on the outside of a pipe mandrel. The microstructure of the HIP'ed OD VPS NARIoy-Z is shown in Fig. 5. Again, the unetched microstructure (Fig. 5a) is very dense. No oxide is visible, and the porosity is less than 1%. The etched microstructure of the OD sample (Fig. 5b) is very similar to the ID microstructure. Hot isostatic pressing was used to increase density and bond strength.

Both ID and OD sprays have minimum porosity \langle <0.2%) after the HIP cycle, with no significant difference in grain size after heat treatment. The intermetallic precipitates are evenly distributed throughout the matrix and grain boundaries for both processes.

4.2 *Microhardness*

Microhardness measurements were made using a Buehler Micromet 1^{TM} (Geophysical Instrument and Supply Co., Inc., Denver, CO) tester with a 100 gf load and a 12 s loading cycle. Microhardness of the as-sprayed and HIP'ed specimens (both ID and OD) was measured at ten locations throughout the thickness of the coating (Table 2). Because the standard deviation of the ID and OD spray microhardnesses is greater than the difference between the averages of each group, no significant difference in microhardness could be determined between the two spray configurations.

43 *Tensile Properties*

Tensile specimens were machined in an axial (longitudinal) orientation from cylindrical and hourglass shapes. Past testing has shown that recrystallized VPS-formed material behaves in a near-isotropic manner. Tensile properties of VPS OD NARloy-Z and wrought NARloy-Z are documented in the literature (Ref 7, 8). The tensile properties of OD VPS NARloy-Z are equivalent to those of wrought NARIoy-Z at room temperature. Ductility at high temperatures (>315 °C, or >600 °F) is less than the wrought material, but tensile strength remains equivalent.

The tensile properties of the VPS ID and OD NARloy-Z are displayed in Fig. 6. The data represent an average of 36 ID and 6 OD sprayed test specimens. Using the same powder chemistry and plasma gun, the ultimate tensile strength of the VPS OD NARIoy-Z is slightly better than the ID NARIoy-Z when tested at 540° C (1000 °F). The ductility of the VPS OD NARloy-Z is equivalent to the ID sprayed material.

5. Discussion

A new method of fabricating near-net shapes has been established. Vacuum plasma spray forming of structural metal components can be accomplished on the outside or inside surface.

Fig. 6 Elevated-temperature (540 $^{\circ}$ C, or 1000 $^{\circ}$ F) tensile properties for ID and OD VPS NARIoy-Z

Long spray durations have produced thick VPS-formed NARloy-Z structures on both ID and OD geometries. Atotally recrystallized microstructure for both ID and OD sprayed NARIoy-Z has been obtained. Combining a high deposition temperature and the HIP process results in a very dense structure for both VPS techniques. The microstructures produced are very similar for both techniques, and there is no significant difference in microhardness. The tensile properties at 540° C (1000 $^{\circ}$ F) of both ID and OD sprayed materials from the same powder lot are essentially equivalent. Monolithic and complex components have been VPS formed in both ID and OD spray orientations. The two processes result in comparable strength and ductility.

The internal VPS parameters were adjusted to produce internally and externally sprayed NARloy-Z with equivalent properties. The net plasma power was increased from 61 to 77 kW to accomplish complete particle melting in the shorter standoff distance required for ID spraying of parts with diameters of less than the required 380 mm (15 in.) standoff distance. Increasing the torch power compensates for the decreased dwell time that the powder particles experience in the shorter flame. The increased torch power also helps to reduce the amount of overspray or trapped unmelted powder found in the deposits.

Higher substrate temperatures of 900 to 1000 $^{\circ}$ C (1650 to 1830 \degree F) are required for ID spraying to generate equivalent recrystallized microstructures. The higher temperature requirement may be the result of the much larger average surface area of the ID parts tested ($>$ 2258 cm², or 350 in.²) compared to that of the OD parts \langle <1290 cm², or 200 in.²) and the long time between successive spray passes, which allowed the coated surface to cool.

Changes in powder injection were required for internal VPS to prevent premature melting of powder inside the plasma gun powder ports. The intense radiated heat from the surrounding part tended to preheat the powder before it entered the plasma. Water-cooled powder feed lines, combined with redesigned powder injection ports, prevented powder from clogging in the plasma gun.

6. Conclusions

- Vacuum plasma spray forming in either an ID or OD configuration has been established for combustion chamber liners.
- Vacuum plasma spray forming using both configurations, followed by hot isostatic pressing, has been shown to produce dense and recrystallized NARloy-Z microstructures.
- 9 No significant difference in microhardness was found for ID and OD sprayed NARloy-Z.
- Long spray durations have produced thick VPS-formed NARloy-Z structures on both geometries.

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