Mechanical and Thermomechanical Properties of Metal Spray Invar For Composite Forming Tooling

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The subject of this paper is the assessment of the thermal and mechanical properties of Invar steel coatings, deposited using electric arc spraying, and the correlation of these properties to the spray parameters and processes used to offer coatings with characteristics appropriate to the requirements of tools used in the fabrication of precision polymer matrix composite work pieces. In particular, two processing methods, inert and air atomization, and three arc spray gun configurations (air cap design) were evaluated. The low coefficient of thermal expansion (CTE) properties of Invar are maintained in the spray-deposited coatings using both high velocity oxy-fuel (HVOF) and air-atomized arc spraying, although HVOF coatings have significantly lower CTE and greater durability than those deposited by arc spraying. The mechanical properties of the coatings are low compared to bulk Invar, regardless of the spray parameters and hardware used. Inert arc spraying affords more consistent coating characteristics but this comes with a compromised durability. The spray hardware was found to be more significant in determining the coating properties than the parameters employed.

Keywords arc spray, autoclave, composite, HVOF, Invar, metal spray, tooling

1. Introduction

The trend toward aerospace projects incorporating an increased number of larger, more complex structural composite components, manufactured to tighter tolerances, coupled with advancements in high-temperature thermoset and thermoplastic resins[1] has encouraged and even necessitated the development of more dimensionally stable tooling. This has been necessary to avoid or minimize the thermomechanical errors that arise from a mismatch in the coefficient of thermal expansion (CTE) between the tool and component materials.[1]

In addition to dimensional stability, the principal manufacturing processes of autoclave and resin transfer molding also require the tool to be durable (increasing tool longevity and reducing tooling costs) and to have a good level of vacuum integrity, criteria that are becoming increasingly difficult to meet using conventional tooling techniques.[2]

These exacting requirements are met with the use of Invar 36. This is a low-carbon austenitic steel having a nominal composition of 36% nickel, a total of less than 1% of other elements (Table 1), and a balance of iron (~64%).[3] A CTE similar to that of epoxy preimpregnated (prepreg) composites (2.2 to 2.9 $\times$ 10^{-6}/°C)[2,4] and good hardness (74 Rb)[1,4] (140 HV annealed to 200 to 260 HV, work hardened)[3] have encouraged Invar 36's acceptance as an aerospace tooling material.

The low thermal mass, short lead time, and economic advantages of composite tooling, combined with the durability of fabricated Invar tooling could offer an optimal solution to these tooling needs.

1.1 Coating Property Measurables

A low CTE is necessary for dimensionally stable tooling and, particularly for large components, is fundamental in avoiding component distortion. The justification for using Invar in autoclave tooling applications is its low CTE, and since the properties of metal spray deposits are often considerably different from those of the bulk material, part of this study is focused on assessing the relationships between the spray process (arc spray and high velocity oxy-fuel (HVOF)) and the CTE. The mechanical properties, namely, flexibility and durability, are also of significant importance to the tooling application. Their relation to the spray process and spray conditions employed, with consideration of the macro- and microscopic properties of the coatings, form the remainder of this study.

The flexibility of the coatings was characterized in terms of the elastic modulus, E, and the ultimate tensile strength, UTS. Determination of the durability of the material is not as easily definable, since this may be influenced by a number of interrelated factors, such as the release coating and fabrication procedure used during component manufacture and the composite/surface interaction. For the purposes of this paper, the macrohardness and microhardness of the coatings were chosen as suitable observables to allow a relative comparison between the coatings to be made. Metal spray coating quality, as viewed at the microscopic level, is quantified in terms of the level of inorganic inclusion and porosity within the coating. These provide measurables that allow the property-structure relationships to be evaluated.

1.2 Metal Spray Variables

The electric arc spray process is fundamentally controlled using five control factors: voltage across the wires (V), current

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Table 1 Elemental composition of Invar 36, controlled expansion alloy

Element	Percentage (at.%)
Ni	36
C	0.03
Si	0.20
Mn	0.30
Fe	Balance

Table 2 Samples prepared for CTE measurement. See text for the composition of the SM8222 powder

Sample	Material	Process
CTE1	SM8222	SmartArc/Air
CTE2	SM8222	SmartArc/N_2
CTE3	Invar (wire)	SmartArc/Air
CTE4	Invar (wire)	SmartArc/N_2
CTE5	Invar SPM4-2242 powder	DJ/C_3H_8
CTE6	Invar SPM4-2242 powder	DJ2600/H_2

through the wires (I), atomizing gas pressure (P), nature of the atomizing gas, and stand-off distance (distance between the spray gun and the substrate). The effect of varying these factors on the coating properties will depend on the specific system and spray materials being used, and, therefore, it would not be sensible to endeavor to obtain a full parameter level-property relationship array. Instead it is the aim of this paper to indicate more general parameter-structure-property relationships that may be used as guidelines to achieve optimum coating quality.

A common problem with the metal spray process is that, because it is a particle additive process, it is necessary to maintain the angle of incidence of the metal spray stream as near to normal to the substrate surface as possible in order to achieve proper consolidation of the coating. Spraying at low angles of incidence results in poor interparticulate adhesion and, consequently, poor mechanical coating properties.[5] Some tooling requirements may have geometries that do not allow a standard arc spray gun access to maintain high enough angles of incidence. Under these circumstances, extension tubes are available to facilitate access to the pattern surface. Since line-of-sight problems are considered important, the influence of the extension tube on the quality of the coatings deposited was assessed.

Table 3 The test matrix employed for the spraying of inert and air-atomized samples

Sample	Air cap(a)	Process gas	Gas pressure (MPa)	Current (A)	Voltage (V)	Spray distance (mm)
1	HV	Air	0.31	100	30	150
1N	HV	N_2	0.31	100	31	150
2	HV	Air	0.41	100	30	250
2N	HV	N_2	0.41	100	33	250
3	HV	Air	0.41	100	25	250
3N	HV	N_2	0.41	100	34	250
4	Fine	Air	0.21	150	25	150
4N	Fine	N_2	0.21	150	30	150
5	Fine	Air	0.41	150	27	150
6	HV	N_2	0.21	200	28	150

(a) Controls atomization
HV = high velocity

2. Experimental Method

2.1 Arc Spraying versus HVOF: CTE Perspective

Samples were prepared and analyzed at Sulzer Metco Application Development Europe (SM ADE, Haltersheim, Germany). As a comparison, Sulzer Metco SM8222 Fe28Cr5C1Mn was sprayed alongside Invar (36Ni, 62Fe, 1.6Nb, 0.4Mn, and 0.2C). The Invar was electric arc and HVOF sprayed, whereas the SM8222 was only arc sprayed. Arc spraying was performed using a Sulzer Metco SmartArc system using 1.6 mm diameter wire. Samples were arc sprayed using both air and inert gas (N_2) atomization. The HVOF application was employed using two different systems, a standard Sulzer Metco Diamond Jet (DJ) propane-fuelled gun and a Sulzer Metco DJ2600 hybrid hydrogen-fuelled gun. Sulzer Metco Invar powder (SPM4-2242) was deposited. Table 2 presents the coatings prepared.

The coating macrohardness was measured using a Wilson 4TT hardness tester. The CTE of the coatings was measured using a Perkin-Elmer (Überlingen, Germany) 7 Series Thermal Analysis System over the temperature range room temperature to 900 °C, with a heating rate of 5 °C min^{-1} in air.

2.2 Arc Spraying versus Inert Arc Spraying

Coating samples were prepared and analyzed at SM ADE using a Sulzer Metco SmartArc arc spray system and Invar wire as above. The coatings were deposited onto 2.5 × 2.5 cm steel substrates using both air and nitrogen as the atomizing gas. The samples were sprayed using a range of parameter levels for current, arc voltage, gas pressure, and stand-off distance. The test matrix employed is presented in Table 3. Sample 5 in the table was sprayed using parameters deemed optimal to obtain a high-quality coating. No equivalent inert arc-sprayed sample was produced.

The macrohardness and microhardness were measured using a Wilson 4TT hardness tester and a Shimadzu HMV 2000 hardness tester, respectively. Inorganic and porosity levels were measured using Image C image analysis software from Imtronic (Berlin).

2.3 Extension-Tube Arc Spraying versus Standard Air-Cap Spraying

Four Invar plates (100 × 100 mm) were sprayed at SM ADE using a SmartArc arc spray system fitted with a PPGT190 extension tube. The tube permits spraying at 90° to the standard direction. Optimized parameters, based on the results of a previous study,[6] were used. The spray parameters used are given in Table 4. These parameters were also used to prepare Invar plates sprayed using incidence angles of 90 and 45°. These were prepared at the University of Warwick, within the Warwick Manufacturing Group, using a SmartArc system fitted with a fine air cap. All samples were air atomized. The coatings were deposited onto polished steel substrates until delamination occurred, so as to obtain free-standing plates.

Table 4 Spray process parameters for the preparation on mechanical test plates

Spray method	Spray current (A)	Spray voltage (V)	Air pressure (MPa)	Spray distance (m)
PPGT 190	195	28	0.41	0.125
45°	200	27	0.41	0.125-0.2
90°	200	27	0.41	0.125-0.2

Table 5 Results for the standard deviation for both air-atomized and inert-gas-atomized coatings

Property	SD(a) (1–4) Air	SD (1–4) N_2
Macrohardness	0.95	2.4
Microhardness	30.3	6.0
Oxide	9.8	1.7
Porosity	0.57	0.14

(a)SD = the standard deviation of the measurements for samples 1 to 4

Tensile and flexural test samples were laser cut from the sheets using a profile as described in BS5600 Part 3: Section 3.7,[7] a standard for tensile test piece geometry for sintered metal materials. Laser cutting was performed at the University of Warwick. Due to technical difficulties involved in thermally spraying test pieces thicker than 1.5 mm, the samples produced did not fully satisfy the dimensional standards for this test. Those standards call for a thickness of 5.4 to 6 mm.

The microhardness, macrohardness, porosity, and inorganic levels were measured as previously described. Tensile testing was performed on an Instron (Darmstadt, Germany) 4505 using a 100 kN load cell. A grip distance and gauge length of 50 ± 0.5 and 32 ± 0.5 mm, respectively, and a crosshead speed of 1 mm min^{-1} were employed. The testing was performed at 23 ± 1 °C and 55 ± 2% Rh. Flexural testing was performed on an Instron 4505 using a three-point bend test jig. The same testing parameters were employed as for the tensile testing. A gauge length of 32 ± 0.5 mm was used.

3. Results

3.1 Arc Spraying versus HVOF: CTE Perspective

The macrohardness and CTE results of samples CTE1 to CTE6 are presented in Fig. 1.

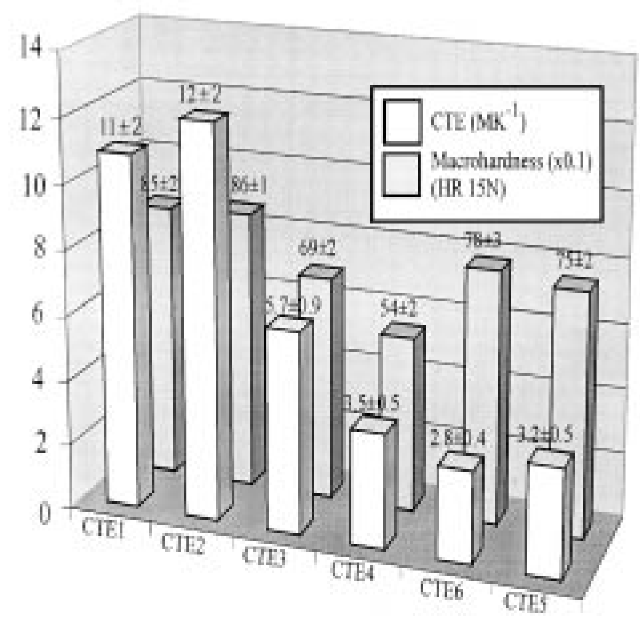

Fig. 1 CTE and macrohardness of arc and HVOF-sprayed 28% Cr and Invar

3.2 Arc Spraying versus Inert Arc Spraying

The property results of the inert and air-atomized arc-sprayed samples are presented in Table 5 and Fig. 2 to 5. Note that results for samples 5 and 6 are not included in the figures since these samples are not equivalent. Micrographs of coatings 4, 4N, and 5 are given in Fig. 6 to 8.

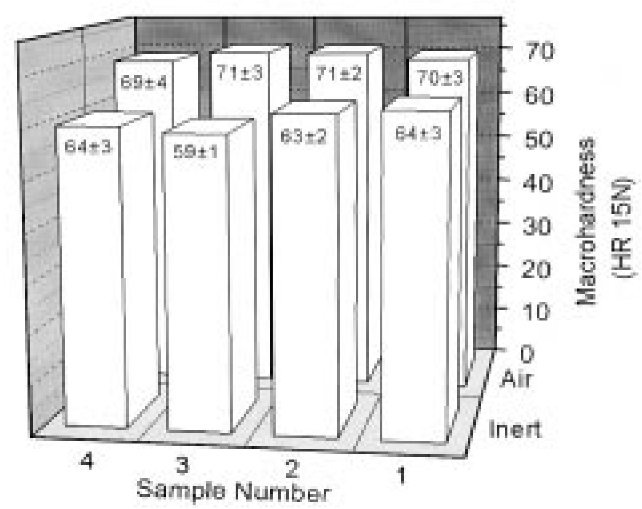

Fig. 2 Macrohardness of inert and air-atomized arc-sprayed coatings

3.3 Extension-Tube Arc Spraying versus Standard Air-Cap Spraying

3.3.1 Young's Modulus Results. The average Young's moduli, Y_{av}, for the samples are presented in Fig. 9 and were determined from the gradient of the linear section of the force-extension plot.

3.3.2 Ultimate Tensile Strength Results. The ultimate tensile strength, UTS_{av}, of the coatings was calculated using Eq 1. The average values for each spray method are presented in Fig. 10.

$$UTS_{av} = \left[\sum^{n} (P_{max}/A)\right]/n \qquad \text{(Eq 1)}$$

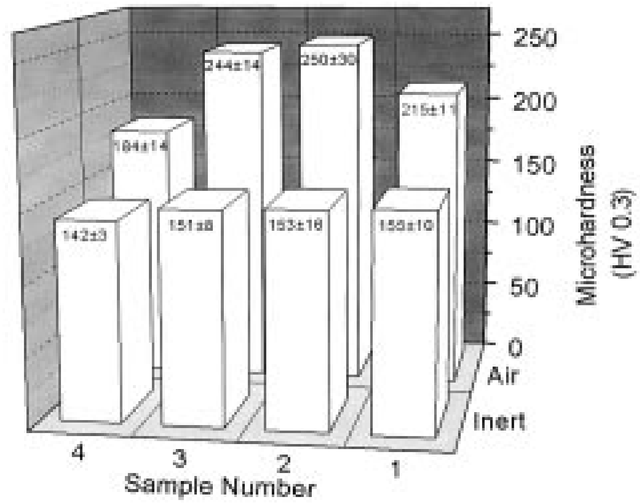

Fig. 3 Microhardness of inert and air-atomized arc-sprayed coatings

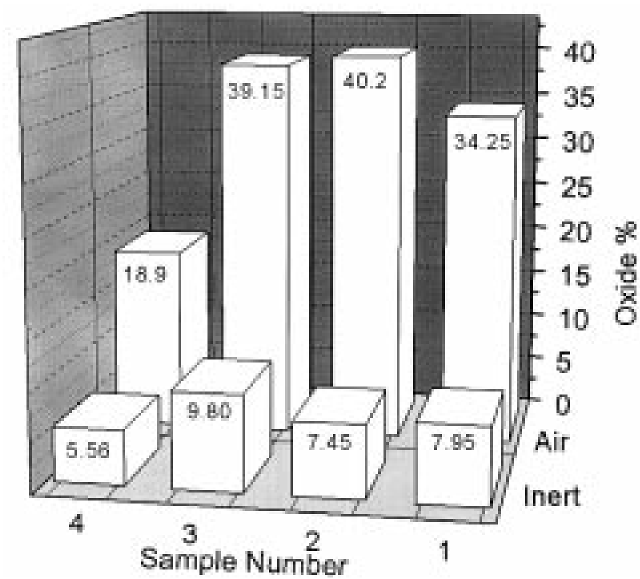

Fig. 4 Oxide content of inert and air-atomized arc-sprayed coatings

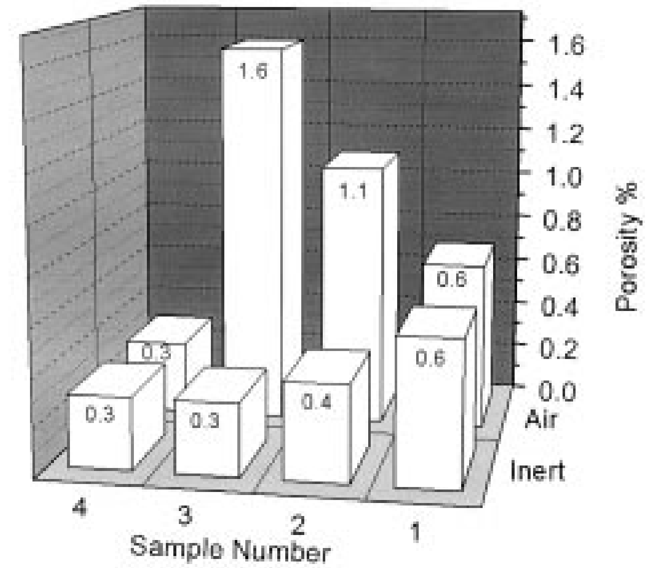

Fig. 5 Porosity content of inert and air-atomized arc-sprayed coatings

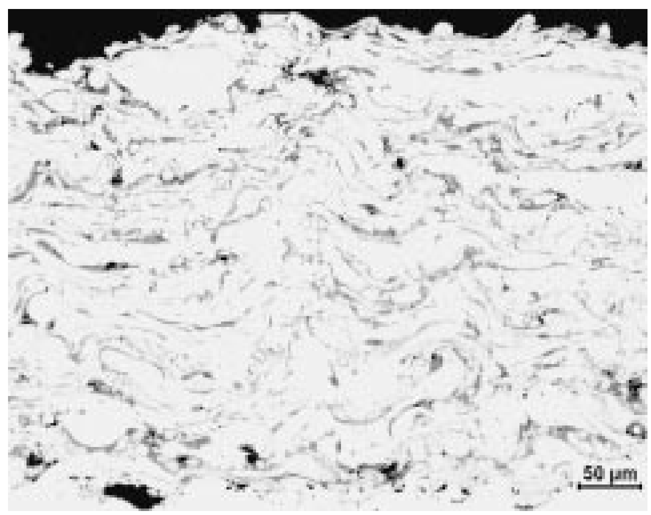

Fig. 6 Micrograph of coating 4 sprayed using air atomization

where

UTS_{av} = ultimate tensile strength (Pa),

P_{max} = force at maximum extension (N),

A = initial cross-sectional area of sample at point of fracture (m^2), and

n = sample set size.

3.3.2 Elastic Modulus Results. The average elastic modulus, E_{av}, was calculated from Eq 2.

$$E_{av} = [\sum^n (L^3/4bh^3) \cdot (F/e)]/n \qquad \text{(Eq 2)}$$

where

L = span length in m (=$32 \pm 0.5 \times 10^{-3}$),

b = sample width (m),

h = sample thickness (m),

F = applied load (N),

e = deformation at applied load L (m), and

n = sample set size.

The value of F/e was determined from a linear regression fit of the linear region of the force-deformation curve for each sample. The results of these calculations are presented in Fig. 11.

3.3.3 Microhardness and Macrohardness Results. The microhardness and macrohardness measurements for the 90°- and PPGT190-sprayed samples are given in Table 6. The 90° sample evaluated for hardness was sprayed for a previous study,[6] but the 90°-sprayed samples for this study were sprayed using identical spray parameters, hardware, and wire.

3.3.4 Porosity and Inorganic Inclusion Results. The

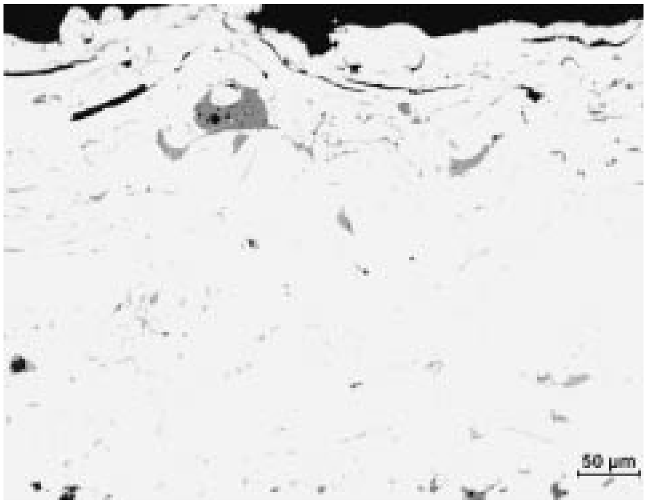

Fig. 7 Micrograph of coating 4N, sprayed using N_2 atomization

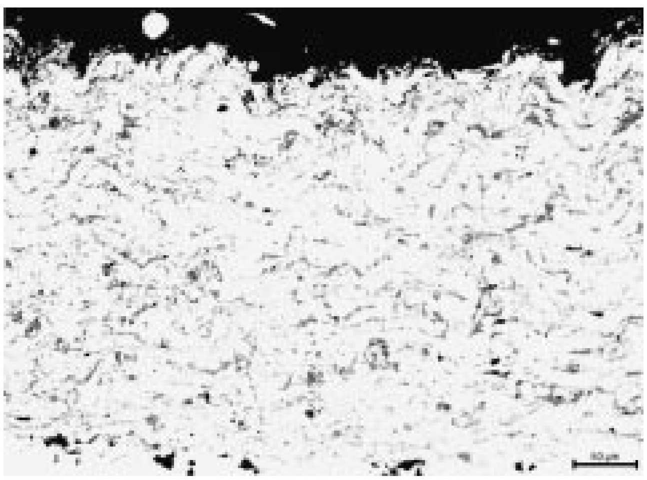

Fig. 8 Micrograph of coating 5 sprayed using air atomization

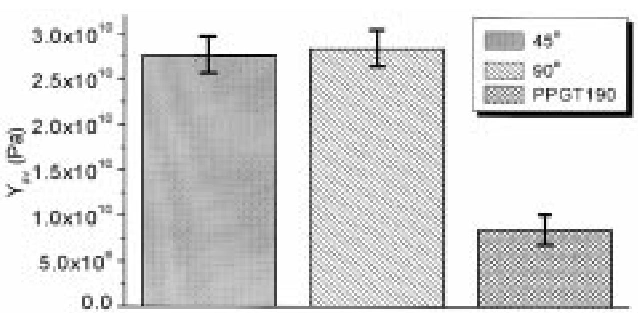

Fig. 9 Average Young's modulus for coatings deposited using various techniques

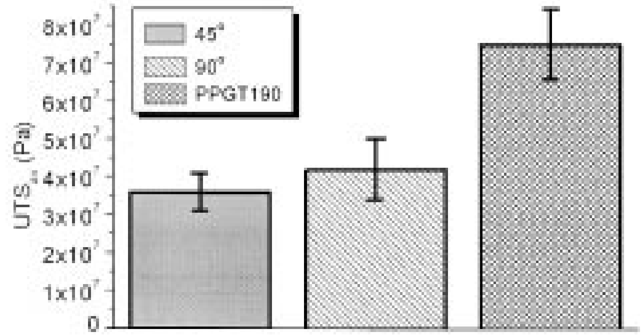

Fig. 10 Average UTS for coatings deposited using various techniques

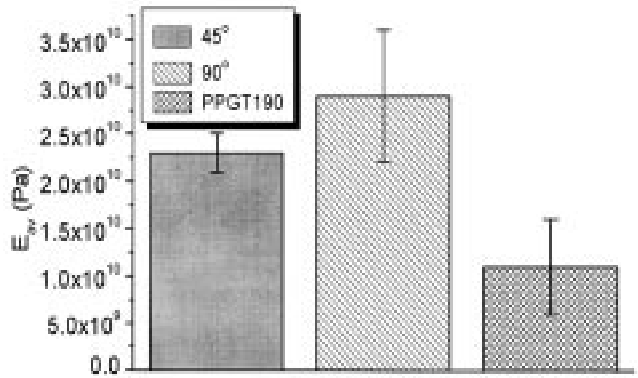

Fig. 11 Average elastic modulus for coatings deposited using various techniques

Table 6 Macrohardness and microhardness for coatings deposited at an angle of incidence of 90° and for coatings deposited using a PPGT190 extension tube

Sample	Macrohardness (HR 15N)	Macrohardness (HV)	Microhardness (HV 0.3)
90°	71 ± 2	238	218 ± 32
PPGT 190	74 ± 1	273	206 ± 12

Table 7 Porosity and inorganic levels for coatings deposited at an angle of incidence of 90° and for coatings deposited using a PPGT190 extension tube

Sample	Porosity %	Inorganic %
90°	0.2	9.95
PPGT190	<1	17

porosity and inorganic inclusion measurements for the 90°- and PPGT190-sprayed samples are given in Table 7. Optical micrographs of the 90°- and PPGT190 arc-sprayed Invar samples are given in Fig. 8 and 12, respectively.

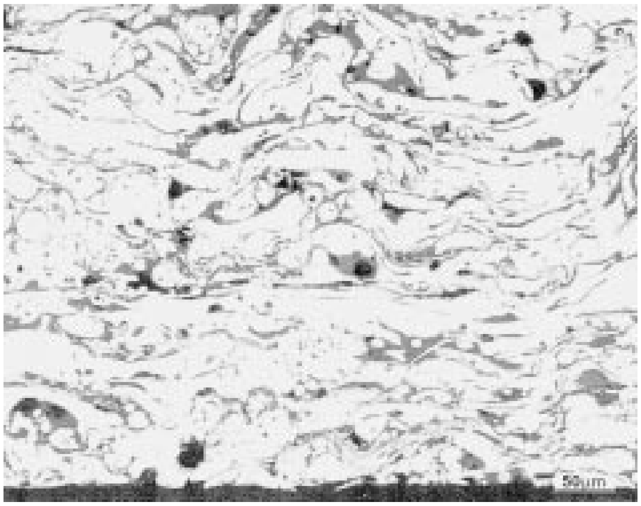

Fig. 12 Micrograph of PPGT190 arc-sprayed coating

4. Discussion

4.1 Arc Spraying versus HVOF: CTE Perspective

The 28% Cr steel (SM8222) was, as expected, the hardest coating evaluated. Both air- and inert-gas-atomized arc-sprayed coatings (CTE1 and CTE2, Fig. 1) have superior macrohardness than even the DJ2600 HVOF-deposited Invar coatings. Although low for a typical stainless steel, CTEs of $11 \pm 2 \times 10^{-6}$/°C and $12 \pm 2 \times 10^{-6}$/°C (CTE1 and CTE2, respectively) are significantly higher than would be required for a dimensionally stable tooling material.

It is clear from Fig. 1 that HVOF-deposited coatings offer the best properties in terms of both hardness and CTE. The HR15N macrohardness values of 75 ± 2 (propane fuelled) and 78 ± 3 (H_2 fuelled) for the HVOF deposits indicate levels of hardness superior to those found even for work-hardened bulk Invar.[2] The CTE values of $3.5 \pm 0.5 \times 10^{-6}$/°C (propane) and $2.8 \pm 0.4 \times 10^{-6}$/°C ($H_2$) are very similar to those of the carbon fiber composite (CFC) component materials (2.5×10^{-6}/°C).[8]

Inert arc spraying offers a very similar CTE to that provided by HVOF, but the hardness of the deposit is significantly lowered to a level similar to annealed Invar.[2] Air-atomized arc spraying offers coatings with good hardness (69 ± 2 HR15N) and a relatively low CTE ($5.7 \pm 0.9 \times 10^{-6}$/°C).

Based purely on the CTE advantage, it could be argued that HVOF is the system of choice for the deposition of Invar for tooling applications. It can be further stated that the added cost of the H_2-fuelled system is not justified. Although the CTE achieved *via* arc spraying is not as low as that obtained with HVOF, it is relatively close to that of the CFC employed in component manufacture. Ultimately, the decision as to which system to employ will be based on the technical requirements of the tooling and the financial constraints of the project, but, considering the economic and user-friendly advantages of the process, it is deemed that air-atomized arc spraying is appropriate for this application.

A compromise between the optimal HVOF and the arc-sprayed coating is the deposition of a hard, low CTE HVOF tool face, backed up with an air-atomized arc-sprayed layer.

4.2 Arc Spraying versus Inert Arc Spraying

From Fig. 4, it is clear that inert arc spraying dramatically reduces the levels of oxides within the coatings, as would be expected. The reduction in oxide inclusions is clearly observed by comparing Fig. 6 and 7 (demonstrated by a significant reduction in the gray inorganic phase). Inert arc spraying also appears to reduce the sensitivity of the spray process to the spraying parameters, *i.e.*, there is a much narrower spread in sample properties. This is emphasized by the significantly lower standard deviation (SD) (1.7 compared to 9.8) for the results of the inert arc-sprayed coatings (Table 5). The change in the level of coating porosity is less significant, but, with the exception of sample 1, where no change was observed, a reduction was observed (Fig. 5).

The reduction in microhardness may be due to the areas of Invar within the inert arc-sprayed coating being larger and more numerous than those in the air-sprayed sample, as seen in Fig. 6 and 7. The microhardness value obtained for the air-sprayed coating is likely to contain elements of both Invar and oxide, thus increasing the value.

The arc voltage is not deemed an important control factor for air-atomized coatings since samples 3 and 4, sprayed under identical conditions apart from voltage, have very similar properties. Similarly, the spray distance does not appear to be a significant factor (within the limits of this study). Samples 1 and 3, despite being sprayed at very different distances, have similar values of oxide content and hardness. The porosity levels, though (Fig. 5), do appear to increase with increasing spray distance. Loss of particle momentum and particle solidification during flight may account for this.

The oxide and porosity levels of samples 4 to 6 are considerably lower than those of samples 1 to 3. This may be, in part, due to the change of hardware from a high-velocity air cap to a fine air cap and also, in part, to an increase in arc spray current. Comparing sample 6 (8.45% oxide) with sample 4N (5.56% oxide), the former being sprayed at a current of 200 A with the HV air cap and the latter at a current of 150 A with the fine air cap, it is inferred that the increase in current is less important in determining the material properties than is the hardware used.

A comparison of samples 4 and 5, prepared under identical conditions apart from atomization gas pressure, suggests that a pressure increase yields a concomitant decrease in oxide level. As would be expected, atomization at higher pressure produces a finer particle size (comparing Fig. 6 and 8). Smaller particles offer a larger surface-area-to-volume ratio, and, since oxidation occurs at the surface, a higher oxide level would be expected for the coating deposited at higher pressure. This is not the case and, actually, the contrary is observed. It is thought that the increased air pressure and smaller particle size result in an increased particle velocity and reduced particle enthalpy, both limiting the particle's exposure to oxidizing conditions. Further work would be required to substantiate this.

Inert arc spraying offers "cleaner" coatings, with more consistent properties than air arc spraying, and, based on the criteria of a superior-quality coating having low oxide and porosity levels, it could be stated that inert arc spraying is favorable to air atomization. It should be borne in mind, though, that fundamental to a material's success as a tooling medium is its ability to endure a full production program without requiring replacement. It can be seen (Fig. 2 and 3) that inert arc spraying consistently reduces the hardness of the Invar. The values obtained for the macrohardness of the air-atomized coatings are commensurate with those of hardened nickel steel, whereas those of the inert arc-sprayed coatings are closer to those of an annealed Invar.[3,9] Inert arc spraying thus has a considerable effect on the macrohardness. It is considered that the decreased durability and higher cost of such coatings negate the benefits of this. Only if the mechanical properties of inert arc-sprayed coatings are significantly superior to air-atomized coatings could they be considered. Further evaluation of their mechanical properties is necessary to determine this. Higher levels of inorganic inclusion may be beneficial to the coating in tooling applications provided that critical properties, such as CTE, are not compromised, which, previously in this study, they have been shown not to be.

4.3 Extension-Tube Arc Spraying versus Standard Air-Cap Spraying

As seen above, the particle additive deposition process, indicative of metal spray, imparts to the materials a degree of porosity and inorganic inclusions. As illustrated in Fig. 12, the oxide forms both sheets between successive layers of deposited metal and actually encapsulates the whole droplet. Being mostly oxide, these inclusions offer a large number of brittle fracture points. This brittle nature is reflected in the low UTS values (35.6 ± 0.5 to 75 ± 9 MPa) for all the metal spray samples. These are one order of magnitude lower than those reported in the ASTM standard for Invar (448 to 552 MPa).[10] Although the UTS of the PPGT deposited coating is higher than that of either the 45°- or 90°-sprayed samples (75 ± 9 versus 35 ± 5 and $41 \pm$ MPa), the value is still much lower than that of the bulk material. The UTS of these materials may further be compromised by the inclusion of a certain amount of residual tensile stress within the structure. This stress, a result of the solidification shrinkage of the droplets, will be partially accommodated within the particle-particle boundaries, thus lowering the UTS. To fully assess the influence that the residual stress has on the UTS and other mechanical properties of the coatings, further trials employing fully annealed coatings are currently underway, the results of which will be reported at a later date.

As for the UTS, the Young's modulus and elastic modulus for all the coatings is very low, being only 6 to 18% of the bulk material value (141 to 148 GPa).[3] It is likely that the apparent low moduli are due to the composite nature of the material. The coatings have a laminar structure, with oxide inclusions between the layers. Each lamination is a composite of metal droplets and inorganic inclusion (Fig. 6, 8, and 12). Movement of these metal splats relative to each other may account for the very low moduli observed.

The microstructure of the 90°- and PPGT-sprayed materials are quite dissimilar, as seen by comparing Fig. 8 and 12. The PPGT coating (Fig. 12) has a greater proportion of large particles. Since both samples were sprayed using very similar process conditions (Table 4), the conditions at the spray head must be influencing the droplet size. Furthermore, since the same metal feed rate and atomizing air pressure is employed for both coatings, it is assumed that the large droplets are formed *via* coalescence of smaller droplets at the spray head, possibly due to more turbulent atomizing gas flow.

Despite both the 90°- and PPGT-sprayed materials having very similar hardnesses (Table 6), porosities, and inorganic levels (Table 7), the Young's and elastic moduli of the PPGT sample are significantly lower than the 45 and 90° samples. The PPGT coating also possesses superior UTS. These differences are inferred to be a result of the microstructural differences between the two materials, and may, in particular, be due in part to the larger volumes of inclusion-free metal in the PPGT-sprayed sample and in part to the distribution of inorganic phases throughout the coating.

Although the differences in mechanical properties between the standard arc-sprayed and PPGT-sprayed coatings are small, they do indicate that the PPGT system can produce coatings that are more flexible and possess a higher UTS than standard sprayed coatings, making them more suitable as autoclave tool materials.

5. Conclusions

Optimal coatings, in terms of CTE and hardness, are obtained using propane-fuelled HVOF metal spraying means. Air-atomized arc spraying, being the most economical process, produces coatings with an acceptably low CTE and good hardness.

In general, the mechanical properties of arc-sprayed coatings are low compared to those of bulk Invar[3,10] regardless of the spray parameters and hardware employed. The benefits of increased flexibility afforded by the low elastic modulus ($<20\%$ of bulk value)[3] are negated by their extremely low UTS,[10] suggesting that it is imperative that tool surfaces manufactured using metal spray techniques are well supported to minimize deformation of the tool surface.

Inert arc spraying offers cleaner, more consistent coatings, but the loss of surface hardness may make this process unsuitable for the manufacture of durable autoclave tooling.

The oxide level and hardness of the coatings are, within certain limits, only marginally influenced by the spray parameters, but are significantly affected by the hardware employed.

More flexible and higher-strength coatings are deposited using the PPGT extension tube, although the benefits of the system are small and relatively insignificant when compared to the gulf between the arc-sprayed and bulk material properties.

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