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Corrosion Study of Cold Sprayed Aluminum Coatings onto Al 7075 Alloy

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Abstract

Aluminum coatings were deposited onto Al7075 T651 structural alloy using both cold spraying and arc spraying. Arc spray coatings were produced using optimized parameters for two atomizing gases, namely air and nitrogen. Cold spray coatings were produced using a low pressure system with air and nitrogen as propelling gases. Six surface preparation procedures prior to deposition were evaluated. Interface quality of as-deposited coatings was investigated by means of fluorescent dye interface penetration technique, bond strength testing and backscattered electron microscopy. Environmentally assisted cracking tests were performed to study the corrosion protection capability of the resulting coatings for structural applications. Micrographs of samples taken before and after cyclic load testing in salt water immersion were compared. The results demonstrated that the Al coatings produced by both arc spray and cold spray provide to Al7075 alloy a cathodic protection against cracking and localized corrosion. However, to obtain such coating properties arc spray technique required advanced surface preparation prior to deposition. For cold spray, the surface preparation has minimal influence on the coating properties thus making this process more advantageous than arc spraying for this application.

Introduction

Material degradation due to fatigue and corrosion are two major factors that contribute to the aging of an aircraft structure. Fatigue is a direct result of aircraft use and the original design life of most aviation platforms is based on fatigue life analysis. However, aircraft structural components made of high strength aluminum alloys can undergo localized corrosion damage due to microscopic heterogeneities in alloys, such as secondary phases. Indeed, numerous corrosion-related failures of Al 7075-T6 structural elements resulting from poor resistance to environmentally assisted cracking have been observed [1, 2]. These components were designed with limited attention to corrosion resistance. A solution to this problem is the replacement of aircraft components manufactured from corrosion-prone alloys with upgraded components designed for corrosion protection, while retaining the original design strength. Among the effective corrosion protection strategies, the use of anodic coatings to provide cathodic protection to corrosion sensitive parts is widely used. Surface engineering by thermal spray technologies offers a broad range of functional and protective coatings to the aerospace industry. However, when temperature sensitive alloys are concerned, such as the Al7075 T651, thermal spraying of protective coatings can be detrimental to fatigue life due to the heat transfer. Furthermore, defects at the coating-substrate interface such as porosities, micro-gap or grits can affect the mechanical properties and, under cyclic load testing in salt water immersion, pitting corrosion can upsurge at the coating-substrate interface [3, 4]. The arc spray technique has been successfully used to produce coatings providing cathodic protection to the Al7075 alloy during environmentally assisted cracking without reducing the fatigue life of the structural alloy [5]. However, to obtain such coating properties the deposition process requires a fastidious surface preparation. In this context, we investigated the possibility to use the emergent cold spray technique as an alternative to arc spray in order to simplify the coating process while providing a protection against corrosion to the Al 7075 T651 aircraft structural alloys.

In the cold spray process, a carrier gas is accelerated at supersonic velocity by passing through a convergent-divergent nozzle. Particles are injected, entrained and accelerated to velocity in the 300-1000 m/s range by the drag force created by the supersonic gas flow. Due to their high kinetic energy, these particles are plastically deformed when impinging the substrate, and, by making either metallurgical or mechanical bond, they form a coating. During the whole cold spray process, the average temperature of particles is significantly lower than the melting temperature of deposited material. This particularity of the cold spray technique offers the possibility for coatings on temperature sensitive materials, which is difficult or impossible with other thermal spray techniques.

Experimental procedure

Substrates

The substrate material consists of 7075-T651 aluminum alloy: 5.6% Zn - 2.5% Mg - 1.6% Cu - 0.23% Cr - 90.7% Al.

Surface Preparation

Six different surface preparation procedures prior to deposition were evaluated:

1. Polished to 1200 grit (P)
2. Grit blasted with 24-grit alumina (GB)
3. Shot peened with 300 μm spherical ZrO_2 particles with Almen 6A intensity. (SP)
4. Shot peened and deoxidized with H_3PO_4 (CE1)
5. Shot peened and deoxidized with HNO_3-HF (CE2)
6. Laser ablation (LA)

Chemical deoxidation

The chemical de-oxidation was performed in order to remove the aluminum native oxide, the hydroxide film and the secondary phases of the Al alloy on top surface. Two chemical deoxidations were performed: (1) deoxidation by a solution of 25% v/v of 85% H_3PO_4 (CE1); (2) deoxidation by a solution of 50% v/v of 70% HNO_3 + 2% v/v of 40% HF (CE2).

The detailed procedure for chemical deoxidation is described in reference [3].

Deoxidation by Laser Ablation

Laser ablation technique was performed using PROTAL® system equipped with four Q-switched Nd: YAG pulsed laser (Laserblast 1000 from Quantel). The experimental procedure is described in details in reference [5].

Coating Processes and Characterization:

Coating of ≈300 μm thick pure Al coatings onto Al7075-T651 alloy substrates were produced with both arc spray and cold spray. The arc spraying was performed using a "Smart Arc gun" from Sulzer Metco with pure Al wires of 2 mm in diameter. Both air and nitrogen were used as atomizing gases. High velocity and cap fine nozzle were used when spraying with air and nitrogen respectively. Stand-off distance was 7.5cm. The gas pressure, arc current and voltage were 240 kPa, 100A and 28V respectively. The cold spraying was performed using a low-pressure cold spray system (SST, Centerline, ON, Canada). For all experiments, the inlet nitrogen gas temperature and pressure were fixed at 400 ^{0}C and 0.62 MPa (90 psi), respectively.

Metallographic Preparation and Examination

The metallographic preparation technique is described in details in reference [5]. Metallographic examination was performed using a field emission scanning electron microscope (FE-SEM) (S4700 Hitachi, Japan) in backscattered electron imaging mode.

Adhesion Strength:

Standard ASTM C-633-99 bond strength tests were performed on test coupons that consisted of 12 mm-thick disks of 25 mm in diameter made of Al7075-T651 alloy glued with epoxy to a 25 mm carbon steel cylinder.

Coating Interface Micro-Gap Evaluation

Fluorescent dye interface penetration (FDIP) measurement was performed, according to the technique developed by S.J. Kuroda et al. [6], in order to characterise the coating substrate interface gap. The test procedure is described in reference [3].

Environmentally assisted cracking (EAC) testing

The approach used for evaluation of the coating performance during environmentally assisted cracking is a method used by Wang et al. [7]. The EAC test set-up is an immersed four point bend beam apparatus that undergoes slow fluctuating load at 0.1 Hz. This type of experimental setup is fabricated according to the Standard Practice ASTM G39-99 for preparation and use of bend-beam stress corrosion test specimens. Rectangular-shape 60.0 x 20.0 x 4.5 mm test coupons were machined in the short transverse direction in order to evaluate the coating protection performance in the most susceptible EAC direction of the Al 7075 alloy. While in operation, the cell contained a four point bending assembly with the sample immersed in 3.5 wt.% NaCl solution kept at 25 °C in air. The applied load was kept under the Ys (503 MPa) of the 7075-T6 alloy and oscillated between 24% and 40% Ys in tension (R=0.6) at a frequency of 0.1 Hz. Although this EAC test uses rather slow fluctuating load, the failure mode validates the environmentally assisted cracking mechanism such as SCC or fatigue corrosion. This approach has the benefit to initiate intergranular cracking in the aluminum alloy with a fast response while maintaining the substrate material under elastic deformation as described earlier [4].

Results and Discussion

Arc Spray Coating Results

The arc spray parameters have been optimized in order to obtain high density coatings with strong adhesion to the substrate. Optimal conditions were used for air and for nitrogen as atomizing gases [3].

Figure 1 shows FDIP measurements and bond strength test values for arc spray coatings with respect to the surface preparation. The FDIP was found to be systematically lower when air was used as atomizing gas for all surface preparation procedures. The adhesion strength values were similar for coatings made with air and with N_2 for surface preparation GB, CE2 and LA. For these deposition conditions the adhesion strength ranged from 35 to50 MPa. However, for CE1 and SP, the bond strength was significantly weaker when of the coating from the substrate as observed on the as deposited sample (Figure 2b). Yet, the Al anodic coating prevented the substrate from any corrosion cracking but some localized corrosion pits have been generated as the one identified on the micrograph. This secondary phase spot plays a cathodic role creating a micro galvanic cell that locally disturbs the polarization made by the anodic coating [5]. The micrograph presented in Figure 3c was obtained from sample produced with deposition conditions N_2 and CE2. No Localized corrosion pit or corrosion cracking can be found across the entire cross-section. This result is typical for sample having good adhesion strength and small micro-gap.

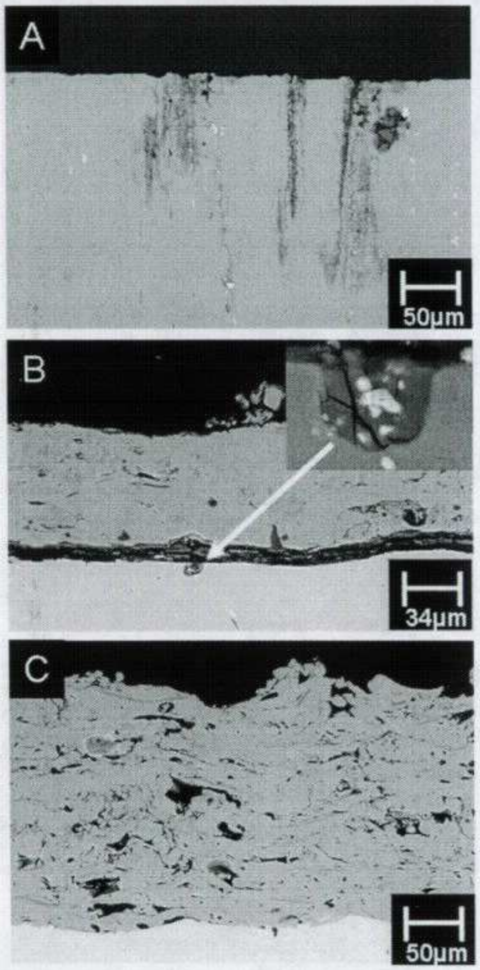

Figure 3 : Cross-section backscattered electron micrograph taken after 30 days of EAC test for sample (a) Al7075 bare substrate, (b) N_2 and CE1, and (c) N_2 and CE2.

The atomizing gas nature has had a critical role in Al7075 alloy fatigue life. Indeed, our previous results have shown that, for Al coating produced with GB surface preparation, the fatigue life is one order of magnitude shorter with air than with N_2. However, the problem with GB surface preparation is the presence of alumina grits at the interface. These defects facilitate the initiation of fatigue failures. The coating produced with air has a secondary oxide phase included which caused defect that are believed to be responsible for the shorter fatigue life observed. The droplets are indeed oxidized during their flight when air is used to atomize the aluminum. The temperature measured on in-flight droplets was found to be around 200 ^{0}C higher when air is used instead of N_2. This difference resulted from an exothermic reaction during oxidation of the particles. To avoid the secondary oxide phase, it is therefore advantageous to use N_2 even if it requires a more complex surface preparation procedure to obtain good bond strength and interface properties. Coatings obtained with deposition conditions N_2 and CE2 or LA conferred a corrosion protection and preserved the fatigue life of to the Al 7075 alloy [5].

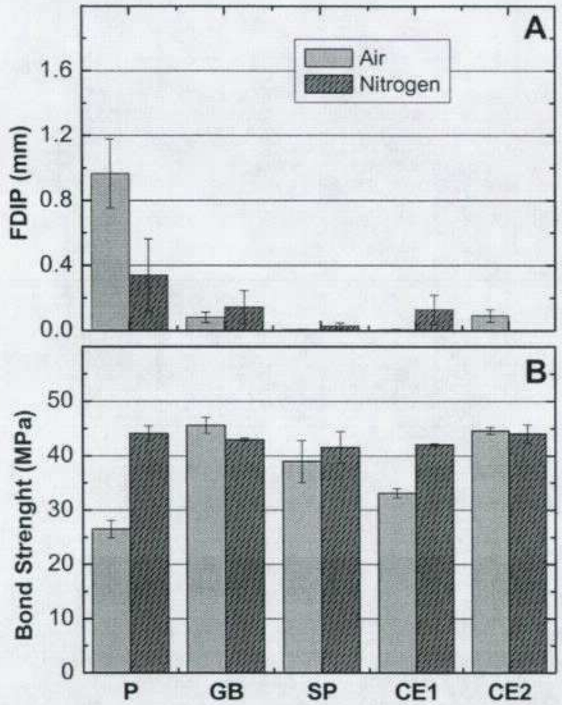

Figure 4: (a) FDIP and (b) bond strength of cold spray Al coatings produced using air or nitrogen as propelling gas with regards to substrate surface preparation.

Cold Spray Coatings Results

The cold spray process parameters and the choice of powder were optimized in order to obtain dense coatings with high adhesion strength. The results of FDIP and bond strength test are shown in

Figure 4. Under nitrogen, the bond strength was over 40 MPa for the five surface preparation procedures. Accordingly, the FDIP were small, ranging from 0 to 0.3 mm. Under air, the bond strength varied to some extent depending on the surface preparation. The values ranged from around 25 to over 40 MPa. For surface preparation P, for which the adhesion was the weakest, the FDIP was about 1 mm. However, for all other

N_2 was used. Indeed, the bond strength value was less than 8 MPa for CE1 and no coating adhesion was obtained for SP with N_2. While the FDIP was small for all conditions using air, surface preparation with GB and LA was required to obtain good results in term of interface micro-gap when using N_2. Cross-section backscattered electron micrographs of as deposited arc spray Al coatings are shown in Figure 2.

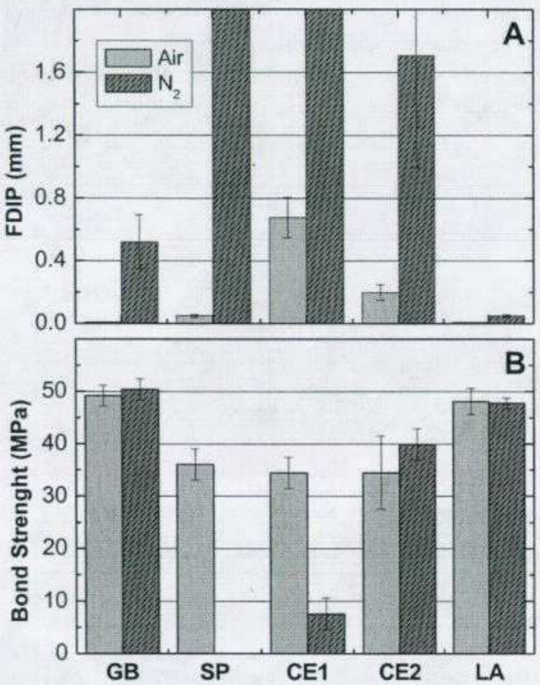

Figure 1 : (a) FDIP and (b) bond strength of arc spray Al coatings produced using air and nitrogen as atomizing gas with regards to substrate surface preparation.

In Figure 2a, the deposition conditions were air and GB, which gave high bond strength and nearly no interface micro-gap.

Alumina grit residues located in the substrate bellow the coating interface can be seen on that micrograph. Beside these defects, the interface quality is very good as no gap or void between the coating and the substrate are observed across the entire cross-section. This micrograph feature corroborates well the results obtained for bond strength and interface micro-gap measurements. In Figure 2b the deposition conditions were N_2 and CE1. The coating is slightly delaminated from the substrate. Although the metallographic preparation procedure might have increased the micro-gap dimension, this result illustrates well the weak adhesion strength quantified previously for that specific deposition condition. Finally, in Figure 2c the deposition conditions are N_2 and LA. Denser coating as comparing to other deposition conditions was obtained using laser ablation performed milliseconds prior to arc spraying. No gap or void can be found at the interface which is in good agreement with previously obtained quantitative results.

Figure 3a shows a typical cross-section micrograph of polished Al 7075 T651 bare sample after 30 days EAC test. The material surface has suffered from intergranular cracking with cracks of about 200 μm long. In most cases, cracks have initiated from localized corrosion. The presence of these intergranular cracking suggests the predominance of SCC since this cracking mode is predominantly intergranular for aluminum alloys [7].

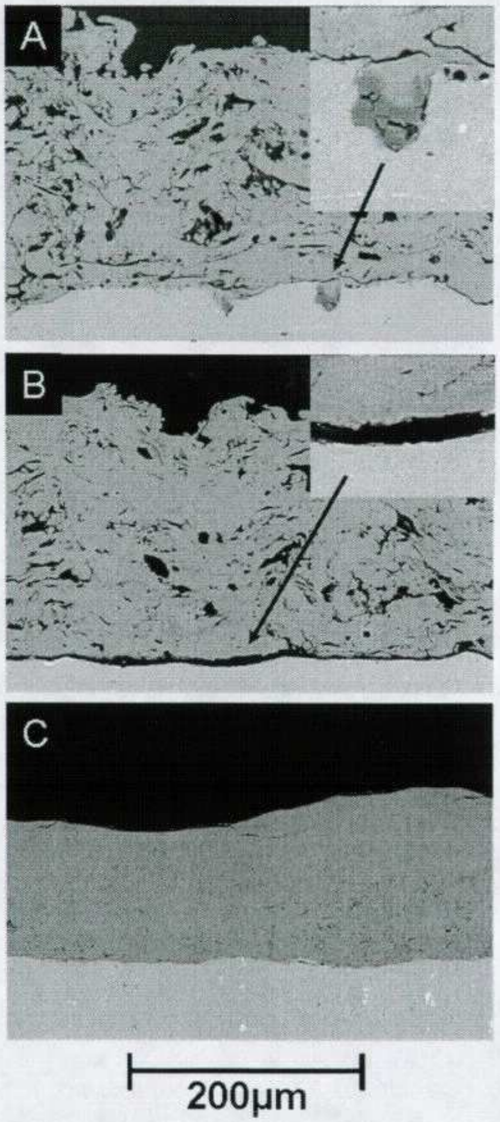

Figure 2 : Cross-section backscattered electron micrographs of arc spray coating with atomizing gas and surface preparation conditions: (a) air and GB, (b) N_2 and CE1 and (c) N_2 and LA.

The micrograph corresponding to deposition conditions N_2 and CE1 in Figure 3b shows partial or localized delamination

surface preparation, the FDIP were significantly smaller, values ranging from 0 to 0.1 mm.
Therefore, except for the surface preparation P, the bond strength and FDIP results obtained with cold spray for all surface preparation and independently of the propelling gas compare well to the best results obtained with arc spray.

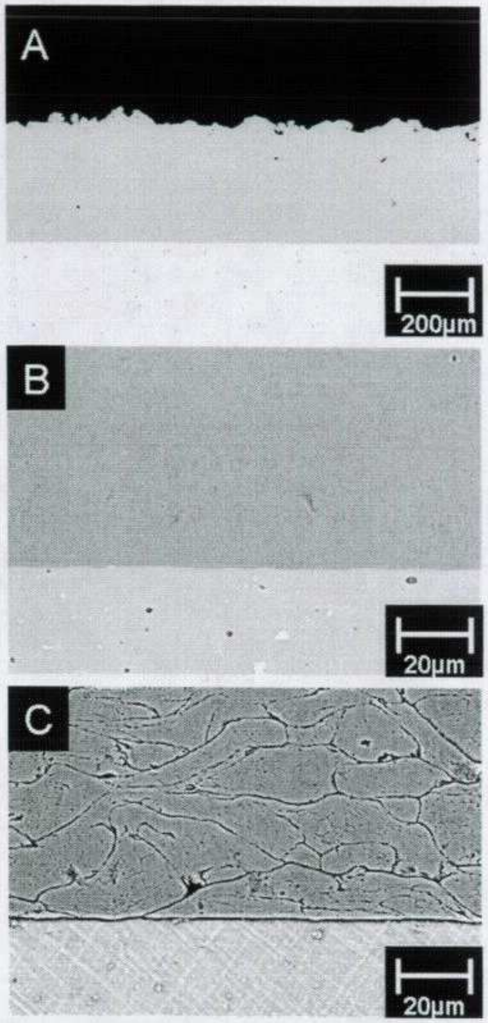

Figure 5 : Backscattered electron micrographs of cold spray Al coating on Al7075 polished substrate at different magnification (a) magnitude 100X (b)magnitude 1000X and (c) magnitude 1000X etched.

For all surface preparation procedures and for both propelling gases, cold spray coatings exhibited very dense microstructures. Figure 5a shows a backscattered micrograph of the coating produced with air and with P as surface preparation. This condition gave the worst micro-gap and bond strength results for cold spray. However, its microstructure is almost exempt of porosity. Even at higher magnification (Figure 5b) no interface defect or micro-void can be found. By etching the sample surface with a Dix-Keller reagent solution, the boundaries between the particles are revealed (Figure 5c). It can be seen that the particles, being spherical in shape before impinging the substrate, have been deformed and flattened. Although no evidence of metallurgical bounding can be seen, it is obvious that a high level of compaction of particles was produced during coating build-up. The porosity was quantified by image analysis and the results are given in Table 1. The porosity was found to be less than 0.5 % for all coatings which is significantly lower than the 3-8% obtained with arc spray [5]. Also, the porosity was systematically slightly lower for air as compared to N_2.

Table 1: Porosity of cold spray coatings with respects to propelling gas and surface preparation.

Surface Preparation	Porosity (%) Air	Porosity (%) Nitrogen
P	0.1 ± 0.1	0.3 ± 0.1
GB	0.0 ± 0.0	0.5 ± 0.2
SP	0.1 ± 0.1	0.3 ± 0.2
CE1	0.0 ± 0.1	0.3 ± 0.2
CE2	0.2 ± 0.1	0.3 ± 0.2

In cold spray, the gas nature influences the velocity of the impinging particles. However, for air and nitrogen, the calculated difference is negligible, as confirmed by our in-flight velocity measurements. Also, the propelling gas was heated to 400 °C but the powder being injected in the divergent part of the nozzle, the transit time of the particles in the hot gas is very short as the gas cools down rapidly when expending. So, we do not expect the particle temperature to increase significantly up to a point when oxidation can occur in such a short delay. Therefore the slight difference in adhesion strength, interface micro-gap and porosity observed between air and N_2 can not be explained by a difference in particle kinetic energy or by oxidation of the particles. . Consequently, the difference between the results obtained for air and nitrogen is most likely due to the experimental uncertainties. Thus there is no clear advantage to use N_2 instead of air for this application

Figure 6 shows typical cross-section backscattered electron micrographs of coatings produced with air for four different surface preparation procedures after 30 days of EAC test. After close examination of the micrographs, we did not notice any difference between as deposited coatings and coatings after the EAC test (under immersion and fluctuating load), neither in the coating nor at the interface. This observation is true for all the surface preparation procedures and for both propelling gases. No cracking and no localized corrosion were found on these samples

These results indicate that cold spray aluminum coatings provide to Al7075 alloy substrate both localized and EAC protection without the need, contrary to arc spraying, of advanced surface preparation such as chemical deoxidation or laser ablation.

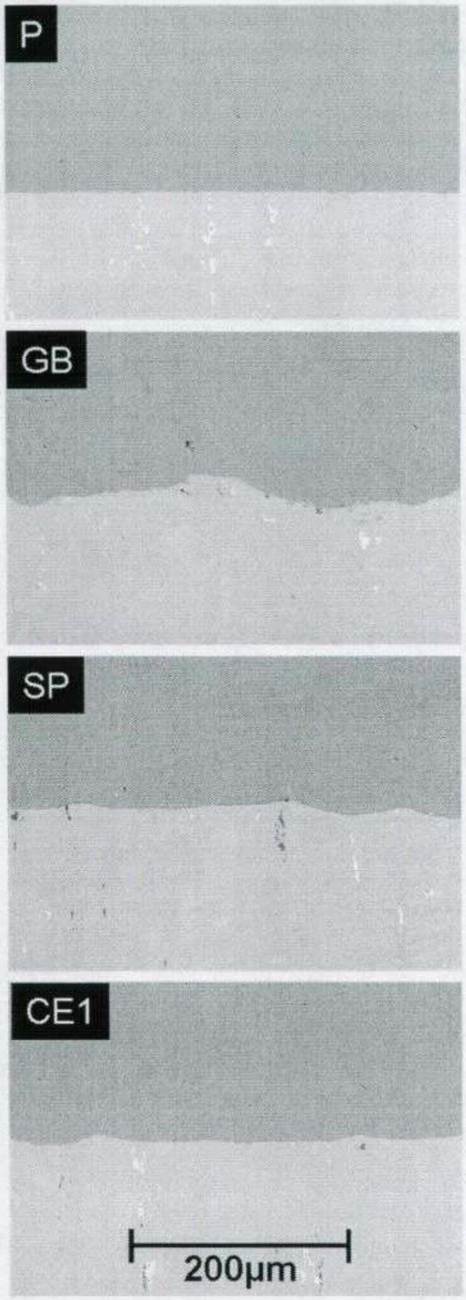

Figure 6 : Cross-section back scattered electron micrographs of air cold spray coatings for four surface preparation procedures.

Conclusion

Very dense cold spray coatings were deposited onto Al7075 alloy using both air and nitrogen as propelling gas. The influence of five different surface preparation procedures prior to deposition was investigated with respect to the adhesion strength, the interface morphology and the cathodic protection against corrosion under environmentally assisted cracking test. The adhesion strength was in the 26-45 MPa range depending on the surface preparation procedure. Coating micrographs examination revealed no gap or void at the interface for all conditions and interface micro-gap evaluation by FDIP corroborated this observation. After a 30-day EAC test, for all deposition conditions studied, no cracking and localized corrosion were found in the substrate. The propelling gas nature between air and nitrogen had no significant influence on the coating properties. Al coatings produced by arc spray also provided to Al7075 alloy a cathodic protection against cracking and localized corrosion. However, to obtain such coating properties advanced surface preparations prior to deposition such as chemical deoxidation or laser ablation were required. Therefore the cold spray process proved to be less complex and more robust than the arc spray process to produce effective anodic Al coating onto Al7075 alloy. Fatigue tests on cold spray Al coatings are underway to complete this study.

Acknowledgements

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