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Aluminum protective coatings - fatigue and bond strength properties with respect to surface preparation techniques: laser ablation, shot peening and grit blasting.

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ABSTRACT

Aluminum coatings can provide galvanic cathodic protection for several metals and alloys. In order to be a suitable protective solution on structural components, the mechanical integrity must be preserved. In particular, the fatigue properties are a challenge for thermal spray protective coatings on mechanical structures. To address the issue of the fatigue integrity of 7075 aluminum alloy with an arc sprayed protective coating, different surface preparations prior to arc spraying were considered. In the present work, a feasibility study was performed using laser ablation as a surface preparation technique before or during arc spraying of coatings through collaboration between the LERMPS laboratory in France, the National Research Council Canada and the Royal Military College of Canada. Both fatigue and adhesive properties of aluminum coatings were evaluated in relation to substrate surface preparation techniques including laser ablation (Protal® process), grit blasting and shot peening. Results indicate that a combination of key conditions including using nitrogen as the arc spray gas, shot peening, and proper laser energy density for ablation, provide high fatigue resistance of metallic coated 7075 alloy substrates. Specimens prepared under these conditions show a similar fatigue resistance to uncoated substrates.

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INTRODUCTION

Substrate surface preparation prior to thermal spraying is a key step to ensure good adhesion of the resulting coating. The most common approach, which consists of two successive stages: 1-surface degreasing by solvent application to remove organic contaminants and 2- surface roughening by grit blasting to ensure mechanical anchoring between the coating and the substrate, faces limitations and drawbacks for certain applications. One drawback is related to the use of solvents that are more and more controlled for health protection and environmental considerations. Another issue is with respect to grit blasting giving rise to residues that can be trapped in the material. These residues create some interface defects that either weaken the coating adhesion or create a severe indentation on the substrate. Both types of defects decrease the substrate-coating fatigue properties to such a degree that it compromises the deposition of protective coatings on structural materials [1,2].

To bypass these disadvantages of grit blasting for surface preparation, the PROTAL® process utilizes a Q-switched Nd:YAG laser for laser ablation of the substrate surface. In this process, the association of the spray gun and a laser “gun” with optical fibers is performed in a particular geometrical arrangement in which the laser treatment precedes by a few milliseconds the deposition of the sprayed material powders. The purpose of the laser irradiation is hence to eliminate contamination films such as grease, oxide layers and hydroxides, to generate a surface state enhancing the deposit adhesion and to limit the recontamination of the deposited layers by condensed vapours from either the thermal spray or laser ablation processes. This process developed by IREPA laser and the LERMPS [3], has emerged from the laser surface cleaning technology where short pulsed lasers (in the nanosecond range) have shown their efficiency in cleaning of metallic [4], organic surfaces [5] and even in art restoration [6]. The laser cleaning principle is based on specific interaction modes [7]. Furthermore, the cleaning efficiency is a compromise between contaminant elimination and integrity of the treated material.

Good results were obtained with the PROTAL® process for plasma spraying applications where the plasma sprayed plume diameter at the substrate surface is not so different from the laser beam area [8]. However, for twin wire arc spraying, the sprayed jet size is much larger than the laser ablated surface. Furthermore, this spraying process generates a significant amount of overspray dust contaminating the substrate surface. Consequently, using the PROTAL® process with the arc spraying process is a new challenge by itself.

This paper targets the following objectives:

1. Evaluate the feasibility of using the PROTAL® process in conjunction with the Twin Wire arc spray process to deposit aluminum coatings onto a 7075 Al alloy.

2. Evaluate the impact of three different surface preparation techniques (Protal, grit blasting and shot peening) for arc spraying with respect to the fatigue properties of the coated part and adhesion of the coating.
3. Evaluate the impact of nitrogen and air as the atomizing gas for the arc spraying with respect to the fatigue and adhesion properties.

EXPERIMENTAL APPROACH

In order to use the PROTAL® process for laser ablation during wire arc spraying, the laser head is integrated in the arc spray gun environment using a specifically designed fixture. This fixture is attached on an ABB 6-axis robot to allow spraying in all directions with the two processes as described in Figure 1. The holding attachment is designed for various laser gun trajectories while taking into account the robot positioning limitations and the limited curvature radius of the SiO₂ fibre optics bundle. Mechanical glides on the fixture allow variations in the relative distance between the two critical process 'spots': the rectangular laser target defined by the optics and the elliptical spray deposit. For optimization of the process, it is also possible to vary independently the size of these two spots by changing the distance of each gun relative to the substrate.

Processing Parameters

Grit Blasting Procedure:

A portion of the 7075 aluminum samples evaluated in this study, were grit blasted with conventional suction type blasting using 24 grit alumina. The grit blasting was manually operated at a distance ≈ 30 cm with an angle of 70° and pressure of 275 kPa.

Shot Peening Procedure:

Shot peening was performed, prior to the coating application and laser ablation, as a surface preparation condition in this study. Zirshot Z-300 ceramic spherical beads manufactured by SEPR in France were used for shot peening. The beads were made of zirconium oxide with particle sizes of $\approx 300\mu\text{m}$ in diameter. The peening intensity was monitored regularly by the use of Almen test strips type A (1.3 mm thick), to induce a 150 μm - deflection as measured with a test gauge conforming to ASE J442. The peening intensity was achieved by 200% coverage. Shot peening was followed by degreasing with an acetone wash.

Laser Ablation:

In this work two Q-switched Nd:YAG pulsed lasers were used for the Protal process (Laserblast 1000 from Quantel) each having an average total power of 40 W. The maximum pulse frequency was 120 Hz with a constant pulse duration of 10 ns at a

wavelength of 1064 nm. The optical beam was guided to the substrate surface through a bundle of eight optical SiO₂ fibres followed with an optical laser head (Figure 1). The beam power density can reach close to $3 \times 10^8 \text{ W/cm}^2$ during the pulse duration. The 8 optical fibres were ranked 2 by 2 to form a rectangle and due to a special optical element inside the laser head, the light intensity was rather uniform in the rectangular beam impacting on the part surface. At the substrate position, the distance between the centre of the laser spot and the center of the arc spray spot was 22 mm in this study. The relative horizontal speed robot/substrate was adjusted in such a way that the repetition rate of the laser impact on the surface corresponded to a 20 % horizontal overlap. On the vertical axis, the increment step size was chosen in order to have a 10 % vertical overlap to secure the total coverage of the surface by the laser beam. The laser-target distances defined by the optical system ranged from 70 to 120 mm to supply energy density varying from 2.0 J/cm^2 to 1.25 J/cm^2 , respectively.

Laser ablation was performed either on polished or shot peened substrates in this study. The polished substrates were repolished to 1200 grit just before the laser ablation in order to remove old hydroxide film. Two laser ablation modes were used in this work:

- laser scouring: consisted of an initial laser ablation of the substrate followed after several seconds by the arc spraying without the inter-layer laser ablation.
- laser de-oxidation: consisted of a laser ablation done either on the substrate after scouring, or on a coating layer during the coating deposition. The geometry of the set-up described in Fig. 1 allows performing the ablation immediately prior the coating deposition with an estimated interval of 0.05 s on flat samples.

Regarding the fatigue samples, because of the two radius curves of the hour glass type fatigue samples, it was not possible to maintain the respective stand-off distance of the Protal and the arc spray processes in one scanning robotic movement. Consequently, the laser ablation and arc spraying were performed successively within 10 seconds apart.

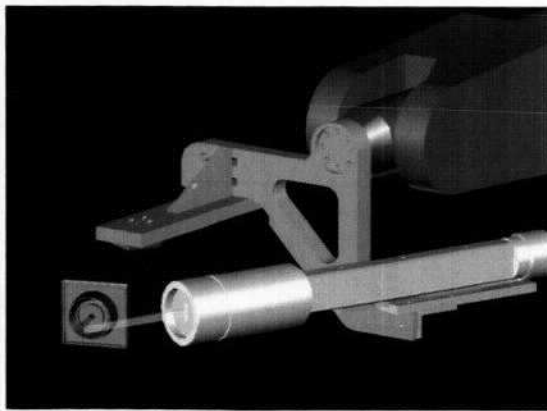


Figure 1 – Schematic view of the optical laser head of the Protal® mounted with a Tafa 9000 arc spray gun on a ABB robot

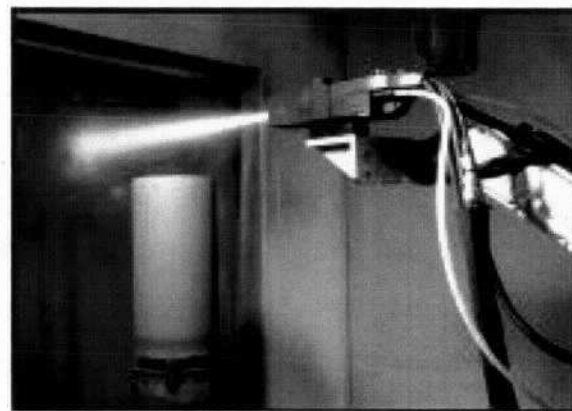


Figure 2 – A typical arc spray process in operation - JP 9000 system shown

Coating Process:

Arc spraying, a thermal spray process for ductile metallic materials, was selected to apply $\approx 200\text{ }\mu\text{m}$ thick aluminum coatings onto 7075-T651 aluminum alloy substrate. The arc spraying uses an electric arc as the heat source to melt two consumable aluminum wires of 2 mm in diameter as illustrated in Figure 2. The melted droplets were propelled onto the substrate surface using two different atomization gases: air or nitrogen. The arc spray equipment selected in this study was a Tafa 9000 gun from Praxair. Coatings were evaluated in terms of their microstructure, adhesion and fatigue properties. The stand-off distance for the arc spray system was selected to produce the smallest spot possible onto the substrate in order to be compatible with the Protal process system. Consequently, the stand off distance selected in this study was 50 mm.

Coating Characterization

Metallographic Preparation and Examination:

The coated samples 60.0 mm x 20.0 mm x 4.50 mm thickness were infiltrated in epoxy resin (Epofix from Struers) then cut with a diamond wheel. The cut section was mounted in epoxy resin followed by a four step polishing preparation:

- 1) samples were ground with SiC 500 grit paper with water; then
- 2) ground with a magnetic grinding disc "Largo" from Struers with $9\text{ }\mu\text{m}$ diamonds and alcohol-based lubricant;
- 3) polished with a magnetic medium hard polishing disc "Dac" from Struers with $3\text{ }\mu\text{m}$ diamonds and alcohol-based lubricant; and finally
- 4) polished with a magnetic soft polishing disc "Chem" from Struers with $0.04\text{ }\mu\text{m}$ colloidal silica and water-based lubricant.

A Hitachi S4700 scanning electron microscope utilizing secondary and back scattered electron imaging (SEM and BSE respectively) was used. The SEM was used to study the cross-sections of metallographic samples and BSE was used to evaluate the porosity and oxide level by the average of eight mapping zones.

Adhesion Strength:

Adhesion tests were performed according to ASTM G633 in order to evaluate the impact of the surface preparation and atomizing gas on the adhesion strength and finally to define if there was a correlation between the coating bond strength and the fatigue properties. The aluminum coating applied on pull test samples was $300\text{ }\mu\text{m}$ thick.

Fatigue Testing:

Cylindrical fatigue specimens were machined from 19.1 mm diameter Al 7075-T651 (UNS A97075; ISO AlZn5.5MgCu) extruded rod. An average minimum gauge diameter of 8.89 mm was machined using a CAD/CAM system over a gauge length of

10 mm. Constant amplitude fatigue tests were carried out at a fully-reversed ($R = -1$) amplitude of ± 225 MPa (stress range = 450 MPa), in accordance with ASTM 466-82. A frequency of 20 Hz was selected to avoid potential frequency-induced heating, with a sinusoidal loading waveform applied via a computer-controlled MTS servo-hydraulic load frame. Testing was performed on coupons prepared with the various surface preparation and coating parameter types described above, as well as on coupons in the smooth and shot-peened states prior to coating. All tests were performed in triplicate, and failure was defined as the incidence of complete separation of the respective coupon halves. The coating applied on the fatigue samples was 200 μm thick and was applied with two arc spray passes.

RESULTS

Metallographic Evaluation

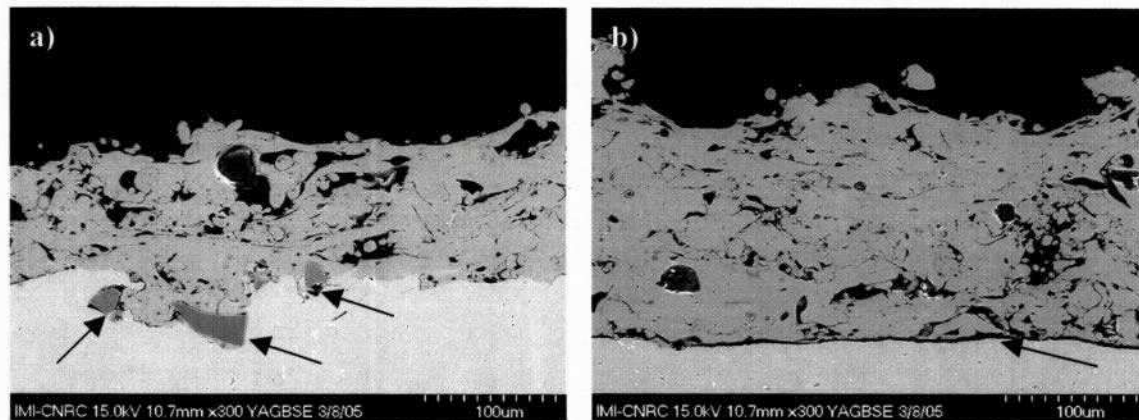


Figure 3 – Cross-sections of an Al arc sprayed coating sprayed using air directly onto 7075 Al: a) on grit blasted substrate with arrows showing embedded grit in the substrate b) on shot peened Z-300 - 6A substrate. The arrow shows a micro-gap at the interface.

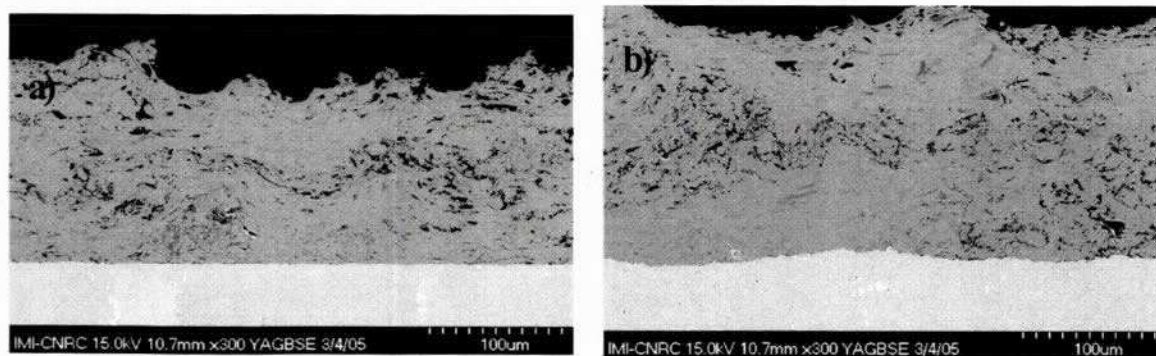


Figure 4 – Al arc sprayed coating; sprayed using nitrogen with laser ablation in scouring mode at $1.5\text{J}/\text{cm}^2$ and de-oxidation mode at $1.5\text{J}/\text{cm}^2$. a) 7075 Al polished substrate; b) 7075 Al substrate as shot peened Z-300 - 6A state.

Figure 3 shows a cross-section of an aluminum coating deposited by arc spraying using air as the atomizing gas and two different surface preparation techniques: a) grit blasting and b) shot peening. Both coatings were applied on a 7075 Al alloy substrate. The coating interface for the grit blasting shows the presence of residual alumina grit at the interface or entrapped in the substrate as indicated by the arrows. For the shot peened surface in Figure 3b), the presence of a micro-gap between the coating and the substrate as indicated by the arrow.

Figure 4 shows the cross-sections of an arc sprayed aluminum coating using nitrogen as atomizing gas and laser ablation on a) a polished substrate at 1200 grit and b) onto a shot-peened surface. It is worth mentioning that the interface was cleaned and in both cases no micro-gap or entrapped grit was observed. Figure 5 shows the resulting microstructures when using laser ablation *in situ* with the arc spraying using air as the atomizing gas. The four microstructures correspond to four different laser energy densities for ablation of the substrate. Figure 6 shows also the resulting coating microstructures when using laser ablation, but with nitrogen as the atomizing gas. Coatings produced by arc spraying processes inevitably contained porosity and oxides.

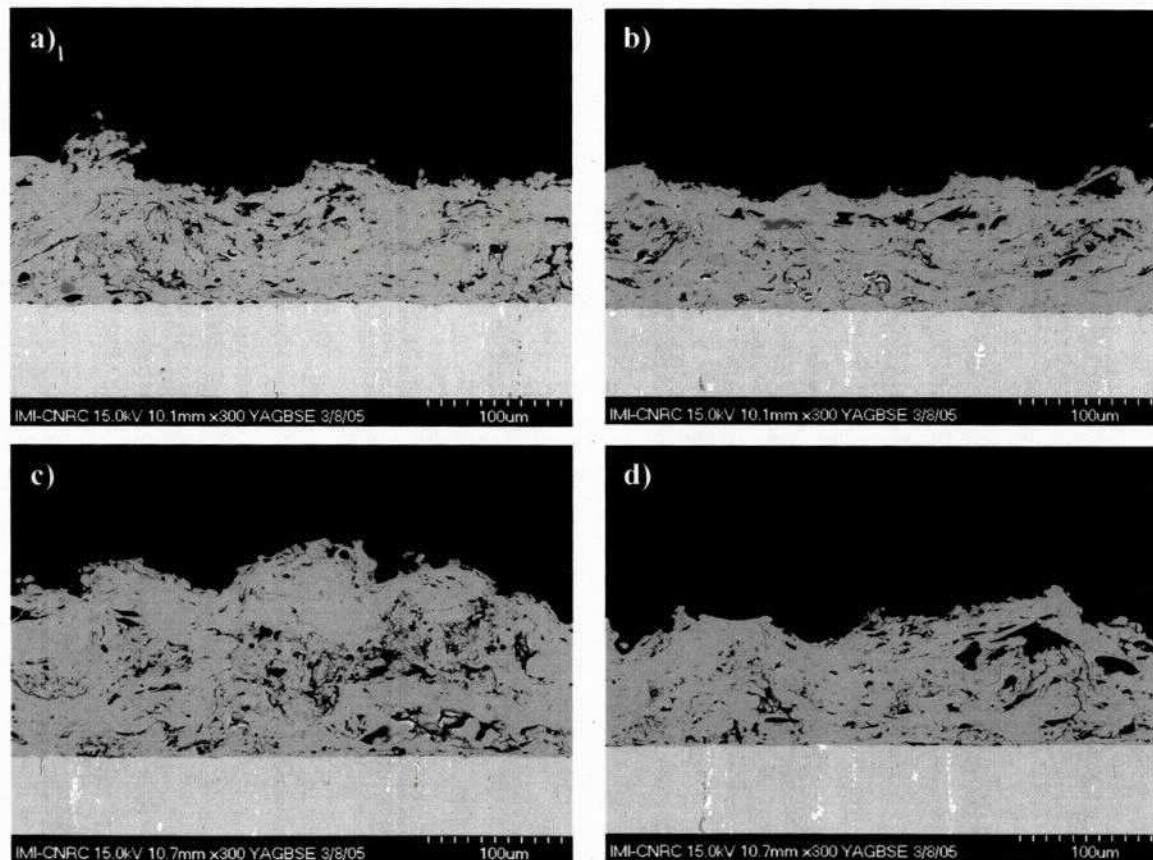


Figure 5 – Al arc sprayed coatings sprayed using air onto 7075 Al polished substrate using four laser ablation conditions performed with 2 passes in scouring mode and two passes in de-oxidation mode: a) 2 J/cm^2 ; b) 1.75 J/cm^2 ; c) 1.5 J/cm^2 ; d) 1.25 J/cm^2 .

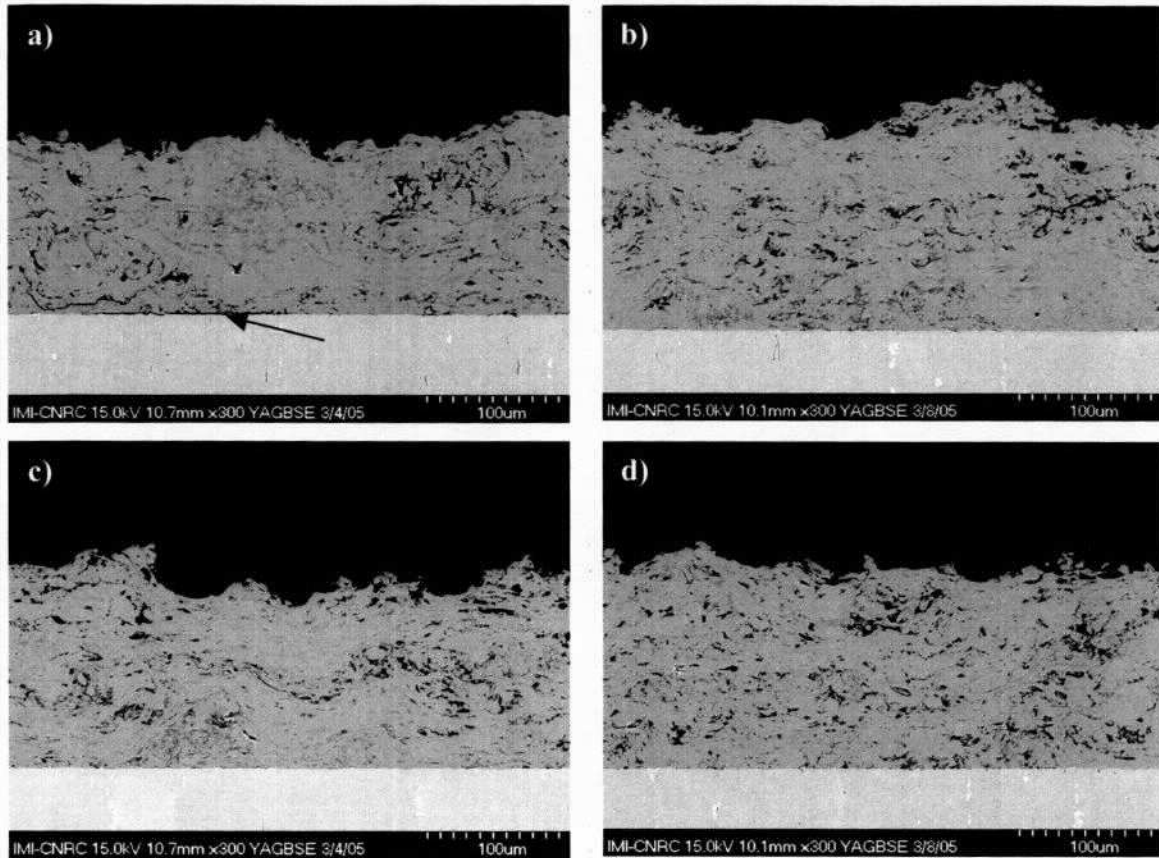


Figure 6 – Al arc sprayed coatings sprayed using nitrogen onto 7075 Al polished (1200 grit) substrate using four laser ablation conditions: a) 2 J/cm^2 in scouring mode only; b) 1.75 J/cm^2 in scouring and de-oxidation modes; c) 1.5 J/cm^2 in scouring and de-oxidation modes; d) 1.25 J/cm^2 in scouring and de-oxidation modes.

It can be noted from Figures 5 and 6 that the use of nitrogen as the atomizing gas reduced significantly the amount of defects such as porosities in the coating. However, some micro-gaps were locally found at the interface for a surface prepared with laser ablation using a laser energy density of 2 J/cm^2 under nitrogen deposition.

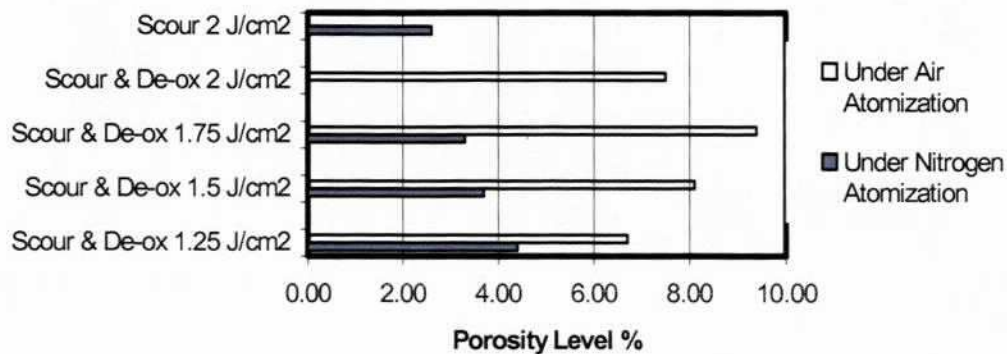


Figure 7 – Porosity level in the Aluminum coating microstructure

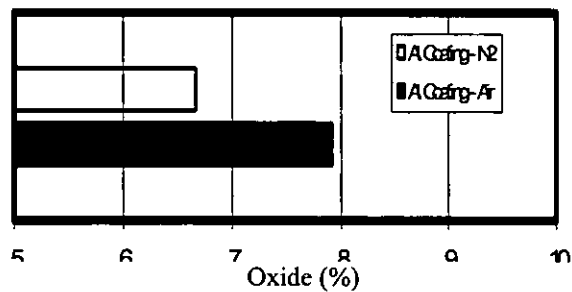


Figure 8 – Oxide level in the aluminum coating microstructure.

A back scattered electron image analysis with the image analysis Visilog software was conducted to determine the extent of porosity and interlamellae defects on the aluminum coating deposited using air and nitrogen with different laser ablation energy densities as shown in Figure 7. It was found that a significantly lower amount of pores and interlamellae defects occurred in Al coatings deposited with nitrogen. Furthermore, it can be noted that the increase of laser power during ablation is favourable to reduce those defects on nitrogen arc sprayed coatings. However, this trend was not observed for coatings deposited with air. Image analysis was also performed to evaluate the oxide level in Al coating microstructure deposited by arc spraying using air and nitrogen, as shown in Figure 8. According to Figure 8 there was a slight reduction of oxides in the microstructure when nitrogen was used as the atomizing gas. However, the oxide level was still non-negligible when using nitrogen during arc spraying because of the presence of ambient oxygen during particle heating and cooling on impact with the substrate.

Coating Bond Strength

Figure 9 shows the bond strength values for the aluminum arc sprayed coating deposited with air onto 7075 Al alloy for four different surface preparations: grit blasting, shot peening, laser ablation on polished and shot peened surfaces with different laser energy densities. The arc spray process using air for aluminum is a robust process in terms of adhesion and allows even an adequate bond strength of 25 MPa when directly deposited on a shot peened surface. Examination of the adhesion results for laser ablation shows similar adhesion to grit blasting in spite of the significantly different interface roughness. Furthermore, the bond strength for coatings deposited with laser ablation on polished or shot peen surfaces is not very dependant on the laser energy density for flat coupons such as pull test or metallographic samples. According to Figure 10, no adequate bond strength was obtained for coatings sprayed directly onto shot peened 7075 Al alloy surface using nitrogen. Furthermore, the average bond strength obtained for nitrogen arc sprayed coatings is 65% of the average bond strength using air in Figure 9. These results confirmed that it is more challenging or difficult to deposit aluminum with nitrogen onto an aluminum alloy and consequently a more optimized surface preparation is needed if high bond strength is required.

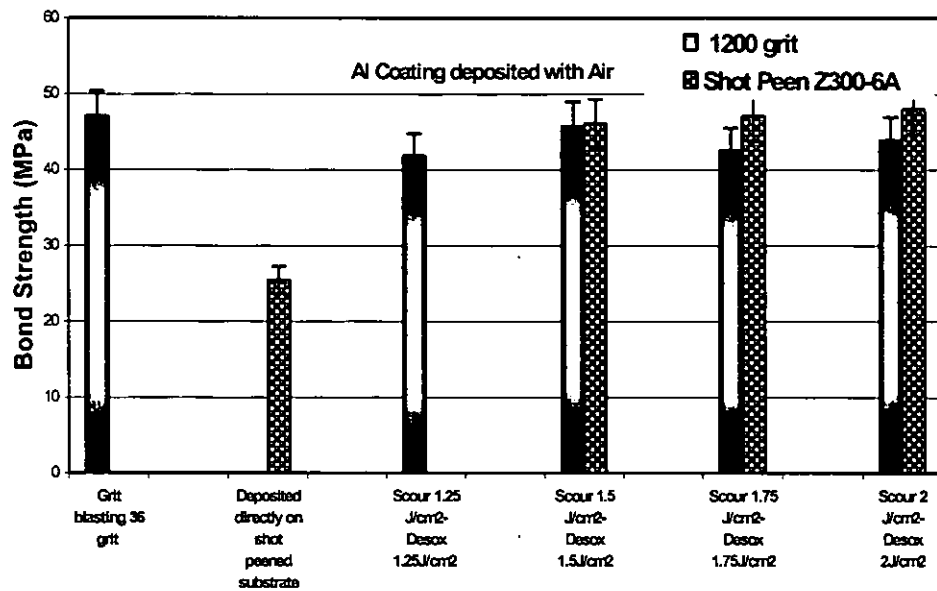


Figure 9 – Aluminum coating bond strength onto 7075 Al alloy deposited with air as atomizing gas

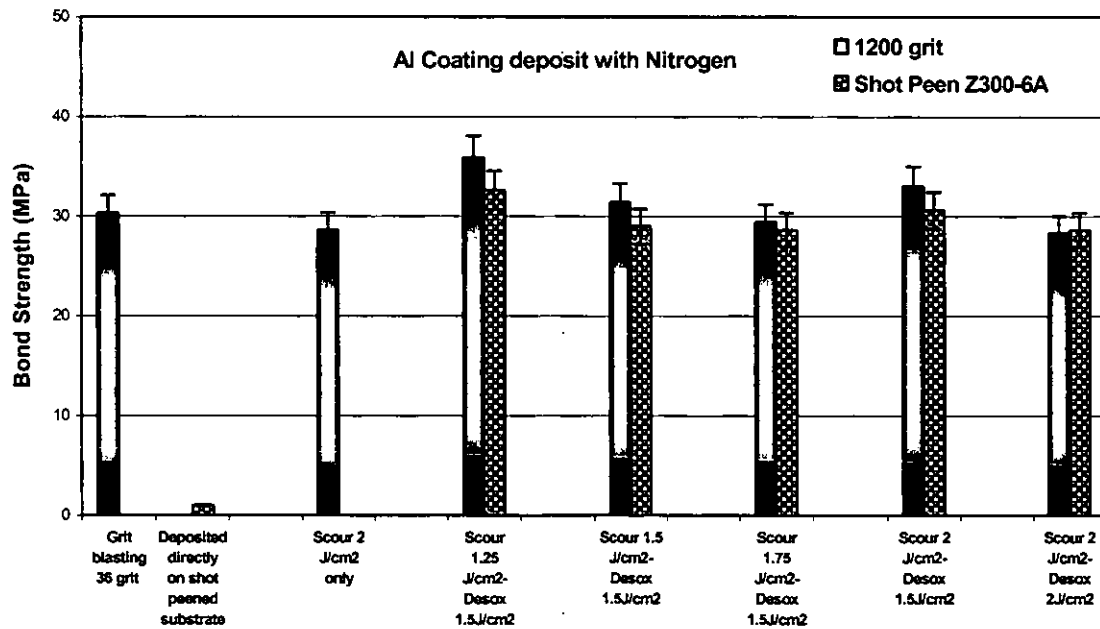


Figure 10 – Aluminum coating bond strength onto 7075 Al alloy deposited with nitrogen as atomizing gas

With respect to the coating bond strength prepared with laser ablation *in situ* with arc spraying using nitrogen, their bond strengths were also similar to those prepared by grit blasting using nitrogen. Examination of the results of Figure 10 indicates that the

adhesion provided by laser scouring on the substrate followed by arc spraying, or laser ablation by scouring followed by a laser de-oxidation and the coating, can provide similar bond strength either on polished or shot peened substrate. Experimentally it was observed that when laser ablation is combined with nitrogen spraying, the dust elimination coming from either the laser ablation or the arc spraying is critical in order to avoid coating spalling.

Fatigue Properties

Figure 11 exhibits the fatigue evaluation of the first two coatings of Figure 9, i.e., aluminum coatings deposited with air onto grit blast and onto shot peened substrates with respect to uncoated 7075 Al alloy. There was evidence of a severe fatigue reduction for both surface preparations and on the 7075 alloy. The fact that the shot peening reduced stress concentration on the substrate (Figure 3), could explain the three fold fatigue life improvement of the shot peened surface over the grit blasted surface. Nevertheless, the shot peened then coated substrates remained at least six times less fatigue resistant than the uncoated substrate.

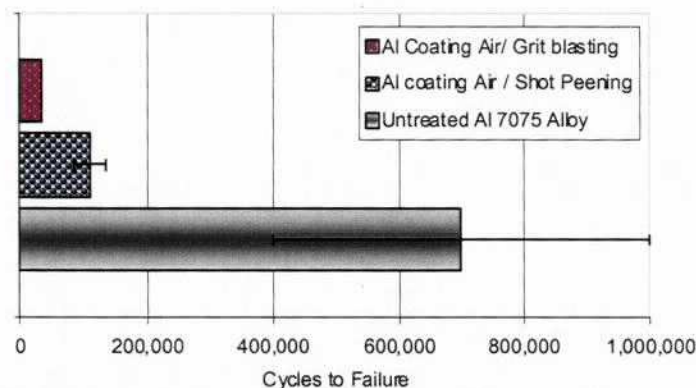


Figure 11 – Fatigue properties of arc sprayed aluminum coating using air as the atomizing gas onto grit blasted and shot peened Al 7075 alloy substrate with respect to the original fatigue properties of Al 7075 alloy evaluated at a stress amplitude of 225 MPa (bars represent 1 standard deviation).

In order to evaluate the fatigue properties of aluminum coating using laser ablation, the laser parameters providing good bond strength in Figures 9 and 10 were applied on fatigue samples. However, the complex shape of the hour-glass shape fatigue samples cause some laser reflection and a lower level of ablation was achieved; consequently coating spalling did occur. Therefore, increased laser energy density on the fatigue samples was used. Figure 12 shows the laser scouring visible marks on fatigue samples for three laser energy densities. Unlike the flat samples, it was not possible to ablate the fatigue samples unless 20 passes at 1.5 J/cm^2 or 4 passes at 1.75 J/cm^2 were performed in order to provide coating adhesion. Only with a laser intensity of 2 J/cm^2 was it possible to perform ablation in only two passes.

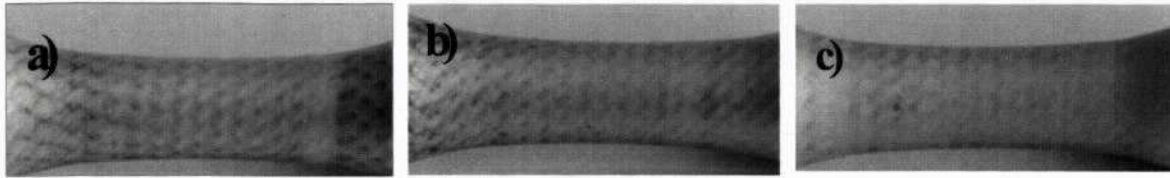


Figure 12 – Laser ablation in scouring mode onto the fatigue sample for three laser energy densities: a) 2 passes at 2J/cm²; b) 4 passes at 1.75J/cm² and c) 20 passes at 1.5J/cm².

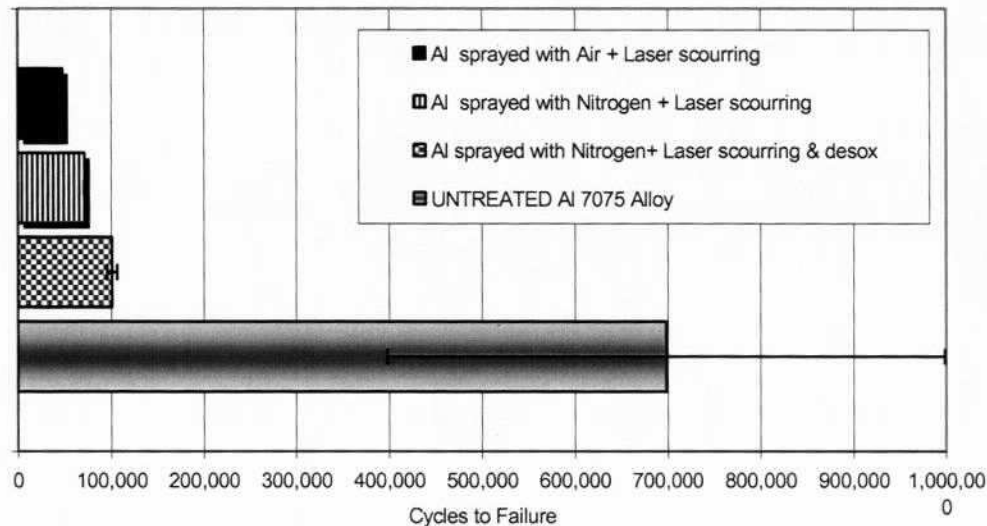


Figure 13 – Fatigue properties of arc sprayed aluminum coating onto laser ablated Al 7075 alloy polished substrate: a) Arc-air + laser scouring 2 J/cm² 2 passes; b) Arc-N₂ + laser scouring 2J/cm² 2 passes; c) Arc- N₂ + laser scouring 2 J/cm² 2 passes and laser de-oxidation 1.75 J/cm².

Figure 13 shows the fatigue performance of aluminum coatings using two different modes of laser ablation: laser scouring and laser de-oxidation. According to Figures 11 and 13, no fatigue property improvement was observed between the use of laser ablation combined with arc spraying using air and arc spraying with air using grit blasting. However, the use of nitrogen and the combination of laser de-oxidation with laser scouring provided a slight improvement in the fatigue performance.

Figure 14 merges the fatigue properties benefit noticed from shot-peening in Figure 11 associated with the benefit of using nitrogen as the atomizing gas as seen in Figure 13 with the laser ablation parameters described in Figure 12. This combination of parameters provided uniform deposition on fatigue samples using scouring and de-oxidation modes with 2 arc spraying passes. In Figure 13, the laser scouring was performed at one laser energy density for the three coatings at 2 J/cm². In Figure 14, the scouring was performed at three different scouring laser energy densities. However, in

order to make the coating adhere to the fatigue samples at lower energy densities than 2 J/cm^2 , multiple passes was required. Although the results showed a response to laser energy, the number of laser ablation passes for scouring is significantly different.

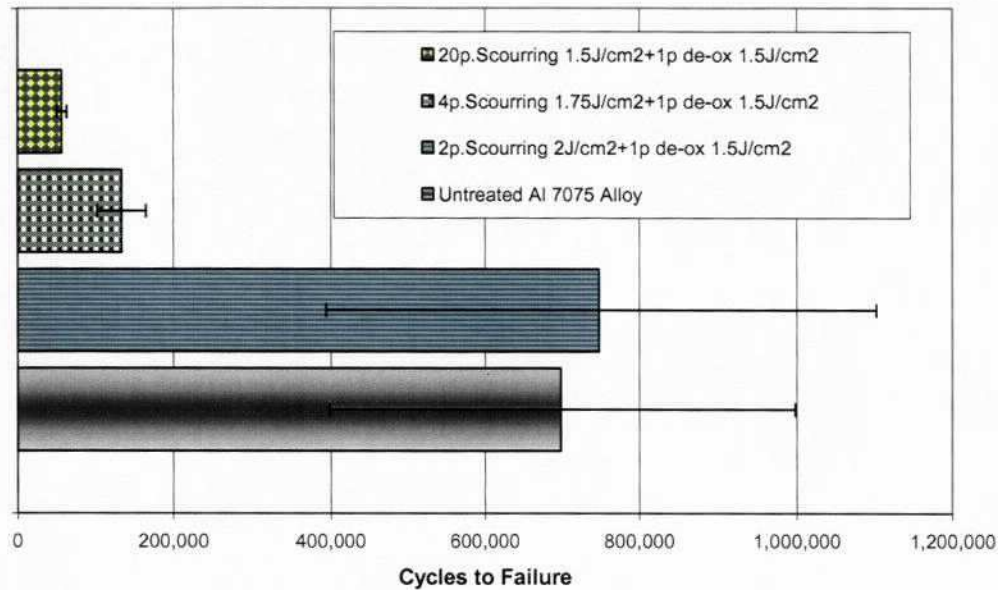


Figure 14 - Fatigue properties of arc sprayed aluminum coating using N₂ onto laser ablated & shot-peened Al 7075 substrate: a) 20 passes laser scouring 1.5 J/cm^2 and 1 pass de-oxidation; b) 4 passes laser scouring 1.75 J/cm^2 and 1 pass de-oxidation; c) 2 passes laser scouring 2 J/cm^2 and 1 pass de-oxidation at 1.5 J/cm^2 with minimum overlapping.

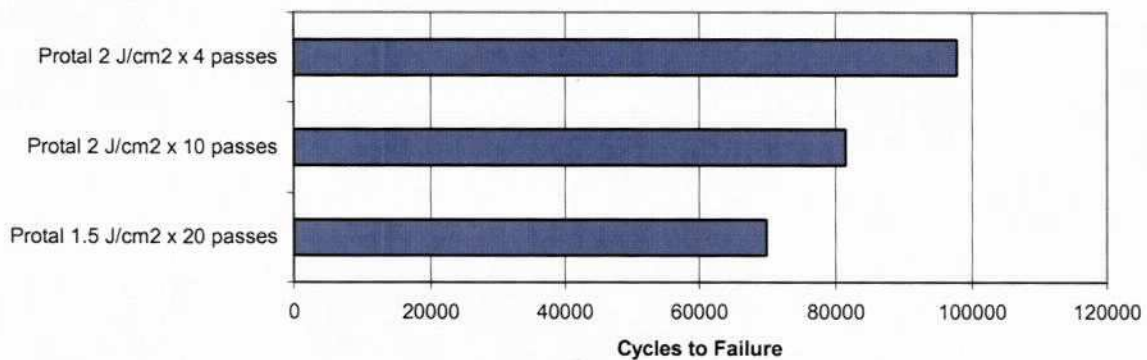


Figure – 15 Effect of laser ablation and overlapping onto the Al 7075 fatigue properties.

In terms of fatigue performance, there is some evidence in Figure 14 that the multiple passes at 1.5 J/cm^2 is detrimental to the fatigue performance. Consequently, in order to evaluate the impact of the laser ablation alone on the fatigue properties, fatigue samples were scoured by the Protal without any deposition of coating and evaluated in

fatigue with the results exhibited in Figure 15. These results suggest that the laser ablation itself reduced the fatigue performance of the Al 7075 and although the repetition of laser passes could improve the adhesion onto complex parts, it remains detrimental in fatigue and therefore the number of passes should be kept to a minimum. Furthermore the number of passes, as described in Figure 12, did increase the amount of oxides on the surface. On a flat surface, it was noticed that on the overlapping between the laser pulses, oxide formation is prominent and dust could be easily form at an excessive laser energy density, and remain on the surface. Such laser ablation dust could be responsible for micro-gaps as shown in Figure 6a. Considering microgaps are an interface defect that can concentrate stresses, overlapping was reduced on fatigue samples when using $2\text{J}/\text{cm}^2$. Indeed the laser spot size was reduced by 50% on the fatigue sample for the test performed at $2\text{J}/\text{cm}^2$.

Consequently, the combination of shot peening and laser ablation in the scouring mode with only 2 passes onto the substrate with arc spraying using nitrogen, provided a breakthrough in fatigue performance with an average fatigue life of 750,000 cycles that is equivalent to the substrate initial fatigue properties, effectively maintaining the fatigue integrity of the polished structural material. Regarding the parameters at $1.75\text{J}/\text{cm}^2$ and $1.5\text{J}/\text{cm}^2$, the number of passes and overlapping were more likely responsible for the reduction of the fatigue properties.

Further work is needed to improve the fatigue properties at lower energy densities with both lower surface overlapping and lower amount of passes in order to reduce the amount of laser ablation dust and oxides at the interface.

CONCLUSIONS

This work has shown that it is possible to thermally sprayed a metallic aluminum coating onto 7075 Al alloy without reducing the fatigue properties. Nitrogen as the atomizing gas for arc spraying process, provides significant improvement for the microstructural density of the aluminum coating. Moreover, laser ablation allows the deposit of aluminum coating onto Al 7075 alloy using nitrogen as the atomizing gas with a clean interface and adequate adhesion. The combination of these two parameters with substrate shot peening has allowed a significant improvement in fatigue properties to the level of an uncoated substrate and consequently maintaining the material integrity fatigue properties for structural applications.

In term of process this study has shown that it is feasible to use the Protal process with the arc spraying process in spite of the large plume and the significant overspray of the arc spray process. Similar bond strength to grit blasting can be achieved with the appropriate laser energy density for scouring and de-oxidation modes. Finally, laser ablation allowed performing a one step process for both surface preparation and arc spraying on a robotized station.

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