

Fatigue behavior of cold spray coatings: The effect of conventional and severe shot peening as pre-/post-treatment

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1. Introduction

Cold gas dynamic spraying, or simply cold spray (CS), is a progressive step in the development of high kinetic energy coating processes. It follows the trend of increasing particle spray velocity and reducing particle temperature in the family of thermal spray methods but to a more extreme level than the high velocity oxygen fuel (HVOF) process. Since the development of the process, a wide range of materials from metals, ceramics and polymers to composites and nanostructured powders have been cold sprayed [1]. The peening effect of incoming high-velocity solid particles in the CS process and deposition at relatively low temperature often results in compressive residual stress unlike traditional thermal spray techniques [2,3]. One of the most important processing parameters in CS is critical velocity. The impact velocity of particles should exceed critical velocity for successful adhesion of the particles to the substrate. Particle temperature, size distribution and

oxygen content can affect the critical velocity value [4–6], the resulting coating porosity, strength and in general the coating performance [7,8].

Considering cold spray as a potential candidate for repairing damaged structural parts is emerging especially in the aerospace industry [9]. Despite the development of advanced composites, the aerospace industry still makes an extensive use of aluminum, titanium and magnesium based alloys. Using cold spray to repair parts made of these materials, can overcome limitations of existing repair technologies (such as tungsten inert gas (TIG) welding and plasma spraying). This is mainly because of the low temperature deposition which prevents melting, development of tensile residual stresses, part distortion and undesirable chemical reactions. The challenge is that the repaired part must retain the bulk material properties to withstand the service loads. Fatigue causes the majority of mechanical failures in service and thus should be fully understood for a reliable design and repair. However, contradictory reports are available on the fatigue behavior of CS coatings of different materials (e.g. Ti [10], Al and its alloys [11] and Mg [12]). Furthermore, the approach typically used to assess the fatigue behavior of CS coated components does not impose a realistic condition on the interface between the coating and the substrate. In this approach, a

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flat, thin specimen, constrained as a cantilever, is subjected to a transversal cyclic loading according to ASTM B593 standard [13]. This method was first used in [14]. The specimen in this method is thin and therefore, a large stress gradient is expected when a bending moment is applied. As the coating thickness is comparable to the substrate thickness in such thin specimens, the interface would be subjected to much less stress than the free surface. Since the interface is frequently the critical point in load bearing, such procedure leads to an overestimation of the real fatigue strength of the coated sample. To represent a more realistic condition, hourglass rotating bending fatigue test specimen [15] is considered in this work. The specimen diameter is two orders of magnitude larger than the coating thickness. Therefore, the in-depth stress gradient effect at the interface is strongly reduced. In fact, the interface is subjected to almost 97% of nominal applied stress in these experiments making a realistic competition for crack initiation at the surface or the interface and thus leading to more reliable results.

The results of the available literature on the fatigue behavior of CS coatings (Both detrimental and beneficial effects) were analyzed in a previous study by the authors and a formula was proposed to predict the fatigue limit of CS coated components. Four principal parameters are determined to contribute to the fatigue behavior of CS coatings. They are interface quality (i.e. adhesion of coating to substrate), residual stress, coating and substrate hardness and stress gradient in the specimen [3].

In the present investigation, the focus is on the enhancement of fatigue life of CS coatings using a hybrid treatment. Hybrid surface treatments are combinations of two or more surface treatments and/or coating processes seeking to combine the advantages of both in a synergistic way. Mechanical and thermo-chemical surface treatments such as shot peening (SP), deep rolling and nitriding can improve the fatigue life by means of developing compressive residual stress and/or inducing surface hardening [16]. Hybrid surface treatments might be capable of producing the best combination of desired characteristics. For instance, combination of severe shot peening (SSP) and nitriding was shown to be able to improve local fatigue strength of smooth steel specimens [16] and to reduce nitriding duration without sacrificing the fatigue behavior and mechanical characteristics [17].

Inspired by the idea of hybrid treatments, the effect of combining SP with CS coating on the mechanical and fatigue behavior of Al 6082 alloy is investigated. In SP, small spherical peening media (shots) are accelerated in various kinds of peening devices to hit a target area and plastically deform surface layers. The plastic deformation during impingements, together with the elastic recovery of subsurface layers, generates compressive residual stresses in the surface layers. Work hardening and roughness alteration are two other important effects of SP. Two standard parameters, intensity and coverage, have been set to ensure the repeatability of the SP process [18]. The peening intensity represents the impact energy level of a shot stream. Peening intensity is characterized by determining the peening effect on a standard test strip. The test strip (Almen strip) and a gage (Almen gage) used to measure the strip's curvature, have been standardized for SP industries. The peening coverage is the ratio of peened area to the total target area expressed as a percentage. Full coverage or 100%, meaning the whole target area has been treated at least once, is the minimum coverage needed to ensure improvement by SP [19]. Coverage higher than 100% can be obtained by multiplying the time needed to reach the 100% coverage. Unusually high coverage of 1000% was shown to induce grain refinement as well as compressive residual stress and work hardening [16,17,20–22]. This process is referred to as severe shot peening (SSP) [16,17,23]. Repeating plastic deformations by high velocity impacting balls generates a large

number of defects, dislocations and interfaces (grain boundaries) and consequently transforms the surface microstructure into ultra-fine grains or nano-structure.

The combination of CS and SP and its effect on the fatigue behavior of hybrid treated specimens has not been studied so far. However, the combination of SP with other coatings and surface treatments such as ion-beam deposition [24,25], high velocity oxygen fuel (HVOF) [26], plasma-electrolytic oxidation [27,28], diamond-like carbon coating [29,30] and chromium electro-deposition [31] has been studied. A survey of the literature shows that the effect of combined surface treatments on different mechanical behaviors at different loading conditions is neither trivial nor always beneficial. Therefore, in this research, the effect of combination of CS coating and SP as well as SSP on the fatigue behavior of Al-6082 is experimentally investigated. In particular, the overall effect of hybrid treatment is evaluated by changing: 1) sequence of the treatments and 2) the severity of SP. The treated specimens are characterized by optical microscopy (OM) observations, residual stress measurements using X-ray diffraction (XRD), and roughness measurements. Rotating bending fatigue tests are performed at room temperature and the fractured surfaces are characterized by scanning electron microscopy (SEM). Based on the results, a critical discussion on the selection of SP parameters and the hybrid treatment sequence (SP/SSP before or after CS) is conducted.

2. Experimental procedure

2.1. Cold spray coating

In the present investigation, aluminum alloy 6082, Migliari Alluminio Srl, Milan, Italy was used for the substrates. This is a medium strength alloy with excellent corrosion resistance. It is known as a structural alloy and has the highest strength of the 6000 series Al alloys. The chemical composition of aluminum alloy 6082 is given in Table 1. The same material is also used as feedstock powder for CS coatings (similar material deposition). Al 6082 powders were prepared by LPW, Cheshire, UK by gas atomization in argon atmosphere. The geometry of the specimen is shown in Fig. 1. Conventional and severe SP were performed before and after CS on the target section of the samples.

The spray deposition parameters are presented in Table 2. The coatings were deposited using a CGT-Kinetic® 4000 commercially available high-pressure CS system equipped with standard type-33 PBI nozzle. Standoff distance was 20 mm and all coatings were deposited with a single pass of the gun. The coating thickness is much smaller than the specimen diameter so that a high stress is applied to the interface (which is the weakest point), reflecting more critical working conditions.

2.2. Shot peening

A pneumatic air blast machine with standard steel shots S230 (0.6 mm diameter), was used for SP and SSP at Peen Service Srl, Bologna, Italy. Air blast SP is a peening process in which shots are accelerated by means of compressed air. In comparison with the other kinds of peening, air blast SP tends to a narrower distribution of impact velocity and mainly perpendicular impacts of media on the treated surface [32].

The peening intensity measured on the "Almen A" strip was 6-8 (thousandth of an inch) for SP and SSP respectively. SP and SSP were performed with 100% and 800% coverage respectively. The primary focus of the present investigation is on structural applications of CS coating and whether the fatigue strength can be increased with the

Table 1
Chemical composition for aluminum alloy 6082.

	Mn	Fe	Mg	Si	Cu	Zn	Ti	Cr	Al
% present	0.4–1	0–0.5	0.6–1.2	0.7–1.3	0–0.1	0–0.2	0–0.1	0–0.25	Balance

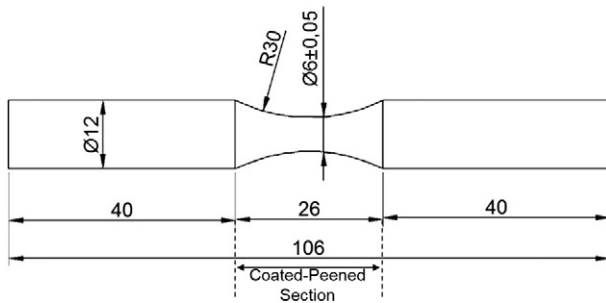


Fig. 1. The specimen geometry used for rotating bending fatigue test. All dimensions are given in mm.

application of SP/SSP as pre- or post-treatment. Therefore, six batches containing 11 specimens per batch were prepared to obtain the fatigue strength. Different batches with corresponding abbreviations are presented in Table 3. The first group is as-received specimens. The second group is CS single treatment. The third and fourth groups were subjected to SP and SSP prior to CS coating. The last two groups were subjected to SP and SSP after CS.

2.3. Coating characterization

Cross sections of the specimens were prepared by a standard grinding, polishing and etching procedure and microstructure observations were performed using Leitz Aristomet, Kirchheim/Neckar, Germany optical microscopy (OM). The samples for OM observations were etched with modified Keller's etchant. To measure residual stresses, XRD analysis was performed using an AST X-Stress 3000 X-ray diffractometer (Cr K α radiation, collimator diameter of 1 mm, $\sin^2(\psi)$ method, the diffraction angle (2θ) of 139° corresponding to the lattice plane (311), tilt angle between -45° and 45°). Xtronic software was used to calculate the residual stress based on the measured strain in the crystal lattice (for more details refer to [33]). In order to obtain the in-depth profile of residual stresses, measurements were carried out step by step by removing a very thin layer of material (around 20 μm) using an electropolishing device. A solution of acetic acid (94%) and perchloric acid (6%) was used for electropolishing. The axial and circumferential residual stresses were measured by X-ray diffraction at the free cylindrical surface exposed by electropolishing. The removal of material can lead to local relaxation of residual stresses. To take the effect of residual stress relaxation into account, the radial stress component was also calculated from an integral of the circumferential stress at the free surface as a function of depth by the method of Moore and Evans [34]. Material removal was carried out until compressive residual stresses vanished (i.e. up to the depth of compressed layer).

A Mahr profilometer PGK Brunswick, Germany (electronic contact instrument) equipped with a MFW-250 Mechanical probe and a stylus with tip radius of 2 μm was used to trace the surface profiles of the treated specimens. The standard roughness parameters were computed based on the acquired signal using Mahr Perthometer Concept 5 software [35]. Surface roughness data were obtained by performing three measurements along three distinct 0.8 mm long surface axial lines. The final reported surface roughness values are the mean value of the three measurements.

Fatigue tests were performed on as coated or as peened condition with stress ratio $R = -1$ and frequency of 20 Hz at room temperature

Table 2
Spray parameters for cold spray coating.

Standoff distance (mm)	Pressure (bar)	Temperature ($^\circ\text{C}$)	Feed rate (Kg/h)	Robot velocity (mm/s)	Gas type
20	30	350	2.5	14	N ₂

Table 3
Specimens' abbreviations.

Group name	Description
AR	As received
CS	Cold sprayed
SP + CS	Shot peening followed by cold spray
SSP + CS	Severe shot peening followed by cold spray
CS + SP	Cold spray followed by shot peening
CS + SSP	Cold spray followed by severe shot peening

using an Italsigma, Forli, Italy rotating bending fatigue machine. The dimensions of specimens used for fatigue testing are presented in Fig. 1. The specimens that passed 3 million cycles were considered as run-out. The staircase procedure (modified Dixon–Mood method) [36] was followed to calculate the fatigue strength. Fracture analysis of specimens was performed by Zeiss EVO50 scanning electron microscopy (SEM) with thermionic source to determine the failure mechanism.

3. Results

3.1. Microscopic observation and surface roughness

SEM observation of the powders is shown in Fig. 2. The powder size, measured by a Malvern ZetasizerNano ZS Dynamic Laser Scattering (DLS) instrument, distribution was between 20 and 63 μm .

The cross-section micrographs of different series are shown in Fig. 3. SP and SSP pre-treatments (Fig. 3 b, c) did not have any significant effect on coating thickness (values in Table 4) as compared to the only-cold sprayed specimen (Fig. 3 a). This shows that prior peening did not change the CS deposition efficiency. SP and SSP after CS deposition (Fig. 3 d, e) flattens the coating particles. Peening post-treatments also spalls the outer layer and produces some cracks within the coating. This side effect is more severe in the case of post-SSP (Fig. 3 e).

Table 4 shows the surface roughness parameters of the specimens. The CS resulted in the highest surface roughness among the studied series. Performing SP as post-treatment considerably reduced the surface roughness due to further deformation of the particles. Pre-treatment by SP also resulted in a slight decrease of the final surface roughness as compared to the single CS process. This is attributed to an increased surface hardness of the substrate by the previous peening treatment, which in turn resulted in higher deformation and flattening of the particles upon impact.

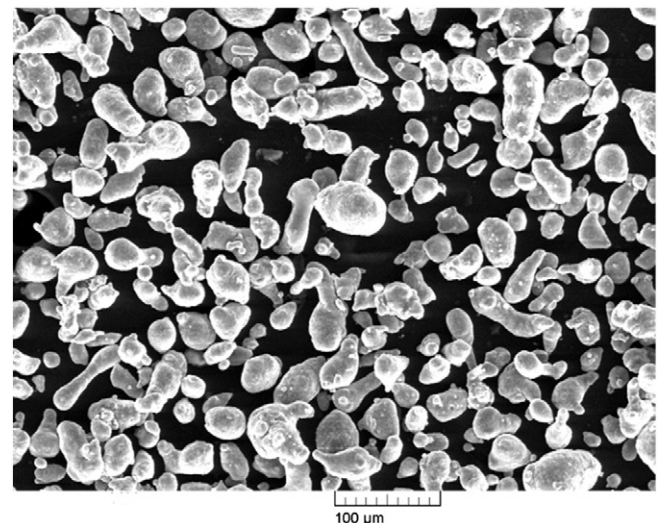


Fig. 2. Scanning electron microscopy (secondary electron mode) of Al 6082 powder.

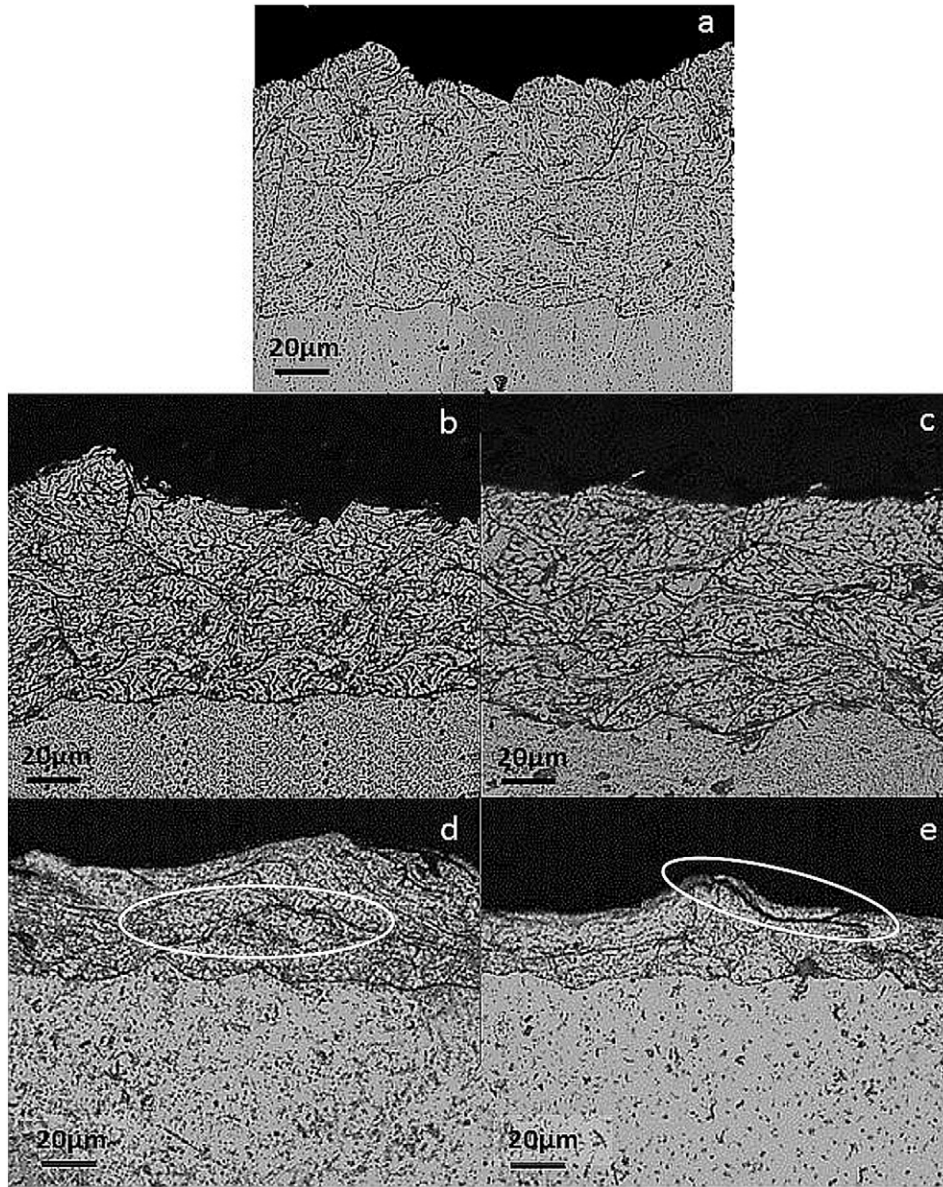


Fig. 3. Cross-sectional micrograph of different treated specimen. a) CS, b) SP + CS, c) SSP + CS, d) CS + SP, e) CS + SSP, and the ovals highlight some damage features after SP/SSP.

3.2. Residual stress

Residual stress profile after peening and CS coating can be characterized by the key parameters schematically shown in Fig. 4. These are: residual stress on the surface, maximum compressive residual stress and the depth at which compressive state of residual stress converts to the tensile counterpart (i.e. depth of compressed layer). No significant residual stress was induced at the top surface, and the measured values for all the series were close to zero. However, significant compressive residual stress was induced in the subsurface layers. Therefore, only the maximum compressive residual stress (which occurred at the substrate subsurface layer) and the depth of compressed layer in the substrate (measured from the substrate surface) are shown in Fig. 4. The maximum value of compressive residual stress is in the same range for different series considering the uncertainty in the measurements. The main difference, however, was found to be the depth of compressed layer for different treatments. Peening before cold spray clearly tends to higher depth of compressed layer as compared to single cold spray or cold spray followed by peening. This will be discussed in details in Section 4.

For comparison, the residual stress development after SP is also taken from the literature. Performing SP on Al 6082-T5 (which its properties especially yield strength is close to the studied material) resulted in the maximum compressive stress similar to the other series (-200 MPa) and the depth of compressed layer equal to $400 \mu\text{m}$ [38] a bit higher than single CS deposition. SSP, generally induces the same level of maximum compressive residual stress but increases the depth of compressed layer significantly [39].

3.3. Fatigue test results

Fig. 5 shows the fatigue strength of the six series as well as the corresponding standard error calculated by the Dixon and Mood model [40]. The staircase test tends to concentrate data near the mean value. Therefore, the method is remarkably accurate and efficient in terms of quantifying the mean fatigue strength. CS itself increased the fatigue strength by 15% as compared to the as-received specimens. Comparing the results of the combined surface treatments show that performing SP/SSP as pre-treatment is more beneficial for the fatigue life improvement. The best result was obtained for SSP + CS which enhanced the

Table 4
Surface roughness parameters and coating thickness after all treatments.

Group name	Ra ^a (μm)	Rq ^b (μm)	Rz ^c (μm)	Rt ^d (μm)	Coating thickness (μm)
AR	0.36 ± 0.01	0.43 ± 0.03	1.92 ± 0.05	2.30 ± 0.06	N.A.
CS	12.41 ± 0.15	15.63 ± 0.17	68.54 ± 2.75	89.86 ± 6.78	80
SP + CS	9.8 ± 0.80	12.5 ± 0.61	53.85 ± 1.69	73.14 ± 0.69	76
SSP + CS	10.9 ± 0.24	13.48 ± 0.25	59.27 ± 3.44	72.29 ± 7.72	80
CS + SP	5.02 ± 0.06	6.35 ± 0.01	26.97 ± 0.02	33.8 ± 1.14	45
CS + SSP	4.74 ± 0.32	5.87 ± 0.14	24.52 ± 0.11	34.85 ± 3.98	35

^a Arithmetic average.

^b Root mean square.

^c Ten-point height.

^d Maximum height of the profile [37].

fatigue strength by 26% and 10% with respect to AR and CS respectively. These improvements are promising given the fact that the tests were performed on smooth specimens with a limited stress gradient.

3.4. Fractography

SEM observation of the specimens after failure are presented in Fig. 6. It can be seen that there are differences in the fracture surface of CS and the hybrid treated specimens. The differences are both in terms of crack initiation and propagation. In the case of CS, the crack initiated at the interface between the coating and the substrate. The boundaries of individual particles, as preferential sites for crack propagation, can be observed within the coating. This shows that intercrystalline crack propagation mechanism is dominant in single CS specimens (Fig. 6b). In contrary, the fractured surface of cold sprayed coatings in the hybrid treated specimens show that the fatigue crack initiated on the surface of the coating and not at the interface between the coating and the substrate (Fig. 6c, d, e, f). In addition, the crack propagated through the coating by combination of transcrystalline and intercrystalline mechanisms. However, the transcrystalline mechanism is dominant in hybrid treatments. Table 5 summarizes the crack initiation point and the dominant propagation mechanism for all the treated specimens.

The boundary between the coating and the substrate is barely visible for the cases where SP was performed before the CS coating (Fig. 6c, d). In these cases, the coating remained attached without any signs of delamination, confirming contribution of the coating to the fatigue load bearing. Application of peening after CS on the other hand, resulted in the separation of the coating from the substrate at the interface during fatigue tests (Fig. 6e, f). This is due to the fact that the coating and the interface were damaged by the peening process leading to eventual delamination during fatigue loading.

4. Discussion

Substrate surface preparation and coating post-treatments can influence coating deposition efficiency, bonding between the coating and the substrate, residual stress and thus affect the resultant fatigue strength.

In the present investigation, the effect of SP and SSP both as pre-treatment and post-treatment on the fatigue behavior of hybrid treated specimens is studied.

SP and SSP as post-treatments (CS + SP and CS + SSP) are discussed first. The peening effect of incoming shots can plastically deform the underlying, previously deposited material. However, the results show that performing SP and SSP after CS are less effective in terms of imparting the residual stress to deeper layers in the material in comparison with the SP and SSP as pre-treatments. This is due to the fact that CS coating is not an ideal continuum and it contains microscopic defects such as non-bonded or weakly bonded interparticles. Therefore, a large portion of the kinetic energy in the subsequent peening is spent to damage the coating rather than inducing an additional work hardening. This can be also observed from the micrographs presented in Fig. 3d and e. Post-conventional/severe SP has also removed some parts of the coating. The material removal could also contribute to the partial relaxation of the residual stress. The failure mode was mainly spalling for CS + SP and CS + SSP. Post-SP and SSP reduced the surface roughness considerably which can be beneficial for retarding crack initiation. However, the damage caused by post-SP/SSP suppressed this effect by producing cracks, and thus no tangible improvement in the fatigue strength was observed.

In conclusion, post SP/SSP treatments triggered delamination, especially when applied by severe parameters. Since the coating has potential macroscopic defects, treatments must be performed with caution in order to avoid damage instead of plastic deformation as in peening of brittle materials [41]. It is worth mentioning that applying post-SP/SSP after a heat treatment of CS coating might be able to improve the mechanical characteristics and specially the fatigue behavior. The heat treatment of CS coating can enhance the ductility by improving the interparticle bonding and thus can reduce or diminish the possibility of damage induced by post-SP/SSP.

In SP/SSP as pre-treatment, the coating remained attached to the substrate during the fatigue loading and showed no signs of delamination. SP/SSP as pre-treatment induced near-surface compressive residual stresses. SSP as pre-treatment resulted in the deepest compressed layer among all the series. This is due to the higher number of impingements

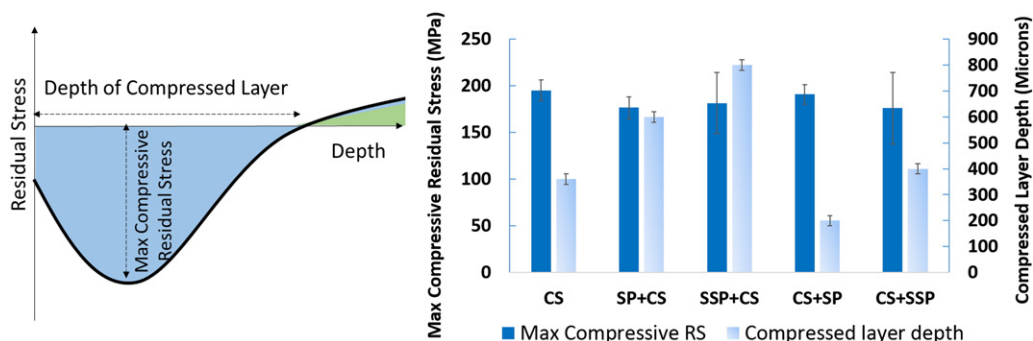


Fig. 4. Maximum compressive residual stress and depth of compressed layer for different specimens.

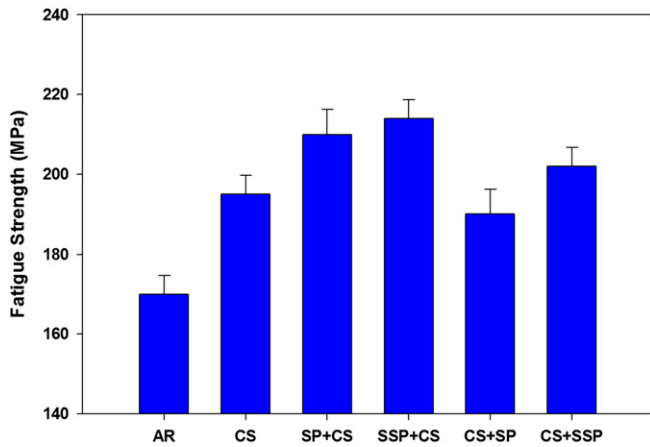


Fig. 5. The fatigue strength of different series.

on the surface points in SSP, which imparts the compressive residual stress to the deeper layers. A good correspondence was found between the depth of compressed layer and the fatigue strength for all the examined surface treatments. The deeper the compressed layer, the higher

Table 5
Crack initiation point and propagation mechanism.

Group name	Crack initiation point	Dominant crack propagation mechanism
CS	Interface	Intercrystalline
SP + CS	Surface	Transcrystalline
SSP + CS	Surface	Transcrystalline
CS + SP	Surface	Transcrystalline
CS + SSP	Surface	Transcrystalline

the fatigue strength; suggesting the critical role of the depth of compressed layer in fatigue behavior of the investigated surface treatments. The compressive residual stress acted as a closure stress on the crack front and reduced the effective stress for its propagation. The high depth of compressed layer conferred protection to areas subjected to higher bending loads.

In this study, the hybrid treatments were not performed immediately one after another. However, if the prior SP/SSP is performed right before the CS deposition, the beneficial effect may increase. Activating the surface and removing the oxide layer at the surface of the substrate without exposure to the air can potentially increase the adhesion strength of the subsequent CS coating and the resultant mechanical properties.

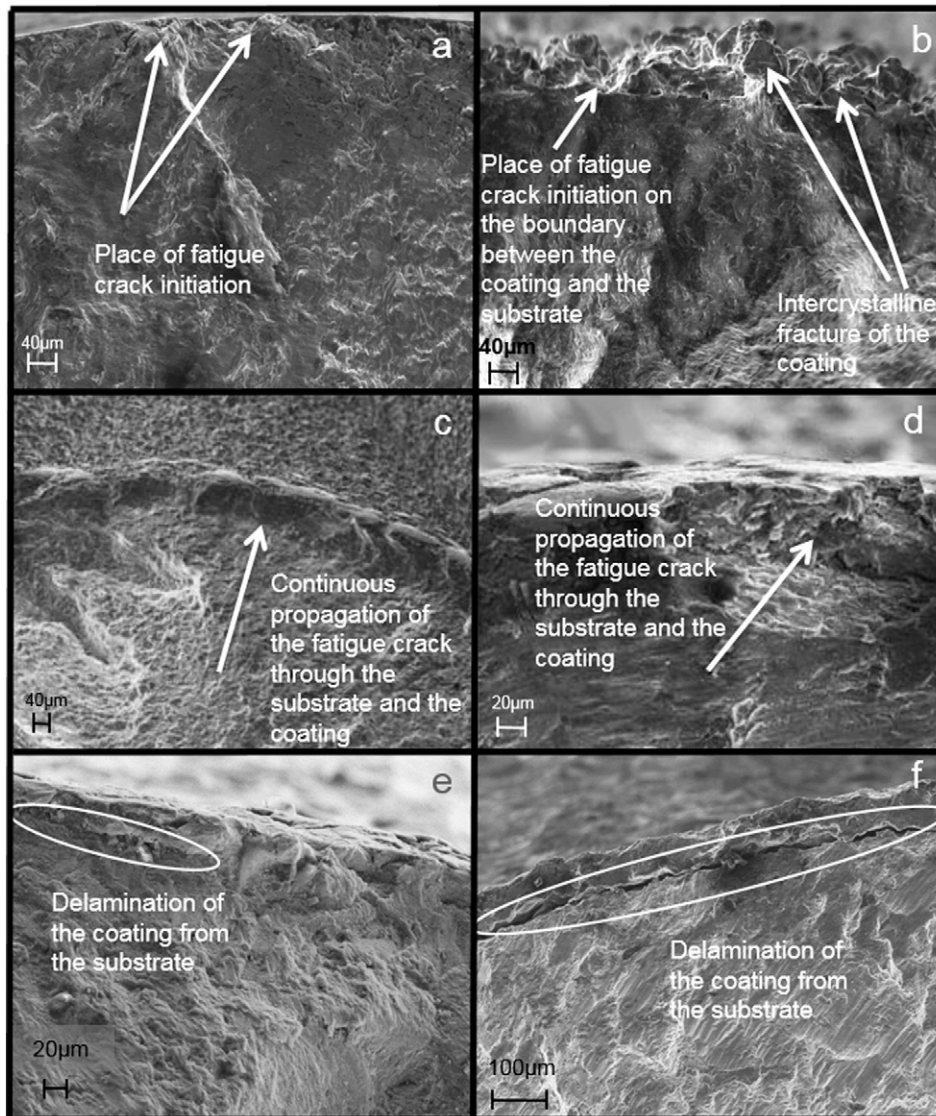


Fig. 6. Detail view (SEM secondary electron mode) of the fracture surface of a) AR b) CS; c) SP + CS d) SSP + CS e) CS + SP f) CS + SSP.

5. Conclusion

The effect of conventional and severe shot peening as pre- and post-treatment on the fatigue behavior of Al 6082 cold spray coating was studied. Hourglass specimens with minimum cross section diameter of 6 mm and thin coatings (in the order of tenth of a mm) were considered to reduce the stress gradient effect at the interface. The cold spray coating itself increased the fatigue strength by 15% compared to the as-received specimen. The conventional or severe shot peening as post-treatment was not able to further increase the fatigue strength of the coated specimens. The peening effect of incoming shots plastically deformed the underlying, previously deposited material. However, since the cold spray coating with the presence of weakly bonded interparticles was not an ideal integrated media, the post-peening could not impart the residual stress into the deeper layers. Instead, the peening caused damage in the coating that acted as initial cracks under the fatigue loading.

It was found that the conventional and severe shot peening are more efficient to improve the fatigue behavior of cold spray coatings if they are performed prior to the cold spray deposition. The severe shot peening before cold spray deposition resulted in a significant improvement of the fatigue strength (26% compared to the as-received specimen). Three main factors contributed to such improvement are:

1. No delamination of the coating from the substrate: The fractography analysis confirmed that the coating remained well adhered to the substrate in the case of cold spray coating with shot peening or severe shot peening pre-treatments. Delamination on the other hand occurred during fatigue loading of the cold spray coating that had been post-treated by shot peening or severe shot peening.
2. Crack initiation and propagation mechanism change: The crack initiated at the surface in cold spray coating pre-treated by peening, whereas it initiated at the interface with stress concentration sites in the case of single cold spray coating treatment. The crack propagation was transcrystalline dominant in cold spray with peening pre-treatment while it was mainly intercrystalline for single cold spray coating. The latter represents propagation through particle boundaries which are weaker due to an incomplete bonding.
3. Residual stress: The depth of compressed layer was higher in the case of cold spray with peening pre-treatment. Compressive residual stress reduced the effective applied stress and retarded the crack propagation.

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