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Number of Passes and Thickness Effect on Mechanical Characteristics of Cold Spray Coating

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Abstract

Nowadays, with severe competitive business environment, limited material sources and high cost of manufacturing, the importance of maintenance and repair is self-evident. In this field, cold spray technology is gaining more and more attention especially in light alloy components. One of the potential applications of cold spray coating is dimensional recovery of damaged structural parts. In most cases, thick coatings are necessary to fill the damages such as cavities, worn or corroded parts. Thick coatings can be deposited in a single or multiple passes giving different thermal input and stress distribution to the substrate and coating itself. The thermal input, the amount and type of residual stress (compressive or tensile) confer appreciable or depreciable characteristics to the coating mechanical properties. In this study, single and multi-pass deposition of a 0.5 mm thick Al 6082 coating on the same substrate is studied to explore the number of passes effect on mechanical characteristics. In addition, one pass deposition of 0.65 and 0.8 mm thick coating is investigated to examine the thickness effect. Micro-structural observation, micro-hardness measurements and X-Ray diffraction (XRD) measurement of residual stress were performed on all groups. Adhesion test and tubular coating tensile test were also carried out to characterize the coating in different cases. Observation of fractured surface was used to investigate the failure mechanism of the cold-sprayed coating. A critical discussion on the effects of pass number and thickness on mechanical properties of coated specimens is presented.

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1. Introduction

One rather recent developed coating treatment that is gaining increasing attention in several industrial applications is cold gas dynamic spray. The cold gas dynamic spraying or simply cold spraying is a progressive step in the development of high kinetic energy coating processes. Similar to the other thermal spray methods in principle, it follows the trend of increasing particle spray velocity and reducing particle temperature as in the high velocity oxygen fuel (HVOF) process, but to a more extreme level. During the process, solid powders (1 to 100 micrometers in diameter) are accelerated in supersonic gas jets toward a substrate. The basic principle of cold spraying has been described elsewhere [1]-[4]. One of the most important processing parameters in cold spray is critical velocity, which particles should exceed to be able to adhere to the substrate. There has been a large effort to investigate cold spray critical velocity. Numerical and experimental studies have been performed to assess the impact process of single particles. Particle temperature, size distribution, oxygen content, nozzle shape and carrier gas type have been shown to affect the critical velocity [5]-[8].

The degree of bonding between particles within cold-spray deposit is also of great importance as it affects the mechanical and physical properties. Adiabatic shear instability and the resulting plastic flow localization are the phenomena that are believed to play a major role in the particle/substrate bonding during the cold spray process.[9],[10].Van Steenkiste et al. [11] described deposition of large aluminum particles ($> 50 \mu\text{m}$) onto a brass substrate by cold spray. They argued that particle melting does not occur and bonding results from severe deformation and subsequent disruption of oxide film on metallic particles allowing nascent metal surfaces to come into contact. Tokarev et al. [12] suggested that particles impacting a substrate in cold spraying first activate the substrate by roughening it. Only once the roughening occurs, the coating is able to initiate and grow. It has also been reported that, with a greater roughening of the substrate surface (going from polished to grit-blasted), deposition efficiency of metallic powders slightly increases [13]. J.G. Legoux et al. [14] measured the coating deposition efficiency as a function of the surface temperature of the substrate during deposition, the gun transverse speed, and the particle velocity. Both single particle impact samples and thick coatings were produced and characterized. Results show that the higher substrate temperature brought about a higher deposition rate of Cu particles, even under the condition where particles were kept at room temperature. Rech et al. [15], [16] showed that the influence of process conditions (gas pressure and temperature, substrate pre-heating, etc.) and the deposition strategy (single pass deposited coatings, multi pass deposited coatings, thickness/pass ratio) is fundamental in the determination of mechanical and microstructural properties of cold spray coating. The presence of tensile stress peaks at interfaces between sequential passes of a multi-pass coating was identified.

One of the potential applications of cold spray coating is repairing damaged parts. Nowadays, with severe competitive business environment, limited material sources and high cost of manufacturing, the importance of maintenance and repair is self-evident. This is even more vital in the case of aeronautical engine, components, frames and large parts where both the production cost and the manufacturing time could be too demanding. Moreover, repaired part must retain bulk material properties to withstand service loads. Conventional repair methods are carried out by thermal techniques on light alloys used in aeronautic (eg. Aluminum and Magnesium). These are very sensitive to high temperature and fast cooling rates. Due to high cooling rates, tensile residual stresses are developed in the material [17], which is detrimental for fatigue behavior. Moreover, tensile residual stress often limits the maximum coating thickness that can be achieved with traditional thermal spray processes. All the above mentioned aspects affirm the necessity of a low temperature-high velocity repairing technique.

It is well established that compressive residual stress and surface hardness are beneficial in terms of fatigue behavior [18]-[21]. The peening effect of incoming high-velocity solid particles in the cold spray process deforms underlying, previously deposited material. This tends to close any small pores or gaps in the underlying material. In addition, the cold spray particles are deposited at relatively low temperature. The net result is that cold-sprayed coatings, unlike most traditional thermal spray coatings, are typically in the compressive residual stress state [22], [23]. Since cold-sprayed coatings generally have no tensile residual stress to drive the opening or extension of cracks in the coating material, most ductile metals can be deposited to almost any desired thickness.

The main concern in the present study, is obtaining thick coatings which are necessary to repair damaged parts. Thick coatings can be deposited in a single or multi-pass, giving different thermal input and stress distribution of the component [16]. The thermal input, the amount and the type of residual stresses (compressive or tensile), vicinity

with air during gun pass and possible oxidation and/or cooling confer appreciable or depreciable characteristics to the coating adhesion and mechanical properties. Therefore, it is important to know the structural properties and peculiarity of respective coating. In addition, coatings with different thicknesses obtained by one single spray pass can also exhibit different mechanical properties.

In the present investigation similar materials for coating and substrate are considered. Coatings have been deposited using high pressure cold spray technique. Single and multi-pass aluminum alloy 6082 coatings of the same thickness are deposited on Al6082 substrate. In addition, single pass deposition of different coating thicknesses is also studied. The treated specimens are characterized by optical microscopy (OM) observation, micro-hardness test, adhesion and tubular coating tensile tests. Residual stress measurement of coating using X-ray diffraction (XRD) is performed on all groups of specimens. SEM observation of the fractured surfaces of the tubular coating tensile test was utilized to explore the failure mechanism of cold spray coatings.

2. Experimental procedure

2.1. Material

The material used for substrate is aluminum alloy 6082, 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. It is a relatively new alloy and is replacing Al 6061 in many applications due to the higher strength. The addition of a large amount of manganese controls the grain structure which in turn results in a stronger alloy.

The same material is used as feedstock for coating to study the effect of similar material deposition on mechanical behavior of treated specimens. This study is valuable for repairing structural parts application. Mechanical properties of Al 6082 are shown in Table 1. Al 6082 powders have been prepared by LPW GmbH using gas atomization in argon atmosphere. Microscopic observation of the powders is shown in Fig 1 emphasizing on the quite spherical shape of the powders with the presence of some agglomerates at the particle shell. The powder size distribution was in the range $-63+20\mu\text{m}$.

Table 1. Mechanical properties of Al 6082

| | Elastic modulus (GPa) | Poisson's ratio | Yield strength (MPa) | Ultimate strength (MPa) | Elongation at break (%) |
|---------|-----------------------|-----------------|----------------------|-------------------------|-------------------------|
| Al 6082 | 70 | 0.3 | 260 | 340 | 11 |

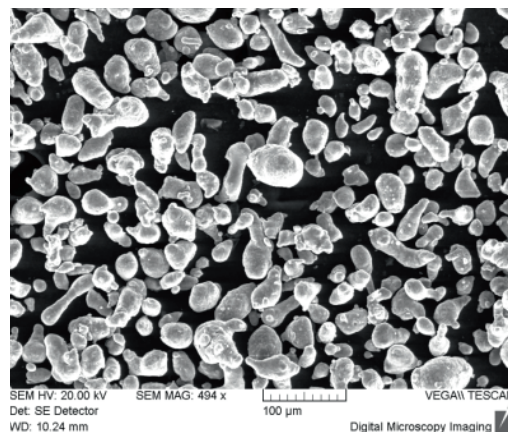


Fig 1. Scanning electron microscopy of aluminium 6082 powder.

2.2. Cold spray coating

There has always been an effort to optimize coating parameters to obtain optimum coating properties [7], [24],[25]. The coatings were deposited at Veneto Nanotech using a CGT-Sulzer Kinetic® 4000 commercially available high-pressure cold spray system equipped with standard type-33 PBI nozzle. Gas temperature has been kept at 350°C as usual with PBI nozzle while gas pressure was approximately 30 bars. Standoff distance was set to be 20 mm. The specimen naming convention with the deposition parameters for different series are reported in Table 2. The first three groups of samples, CS-1P, CS-2P and CS-3P, have the same thickness, but different number of passes for deposition was applied to build up that thickness. The fourth and fifth, CS-T2 and CS-T3 groups together with the first group CS-1P have different thicknesses but they are all deposited in one pass. Two kinds of specimen for tubular coating tensile test and bond strength test have been considered according to the standard test methods [26], [27].

Table 2. Spray parameters for cold spray coating

| Group name | Average coating thickness (μm) | Number of passes | Standoff Distance (mm) | Pressure (bar) | Temperature ($^{\circ}\text{C}$) | Robot velocity (mm/s) | Gas type |
|------------|---------------------------------------------|------------------|------------------------|----------------|------------------------------------|-----------------------|----------------|
| CS-1P | 500 | One | 20 | 30 | 350 | 0.8 | N ₂ |
| CS-2P | 500 | Two | 20 | 30 | 350 | 1.6 | N ₂ |
| CS-3P | 500 | Three | 20 | 30 | 350 | 2.6 | N ₂ |
| CS-T2 | 650 | One | 20 | 30 | 350 | 0.6 | N ₂ |
| CS-T3 | 800 | One | 20 | 30 | 350 | 0.5 | N ₂ |

2.3. Coating characterization

Cross sections of the samples were prepared by a standard grinding, polishing and etching procedure. Microstructure observations were performed using optical microscopy. Specimens for observations have been etched with modified Keller's reactant. To study the state of residual stresses, XRD analysis was performed using an AST X-Stress 3000 X-ray diffractometer (Cr K α radiation, irradiated area of 1 mm diameter, $\sin^2\psi$ method, the diffraction angle (2θ) of 139 $^{\circ}\text{C}$ corresponding to the lattice plane (311) scanned between -45° and 45°). Measurements were carried out step by step by removing a very thin layer of material using an electropolishing device in order to obtain the in-depth profile of residual stress. A solution of acetic acid (94%) and perchloric acid (6%) was used for electropolishing. Material removal has been carried out on the coating up to reaching to the substrate.

The coating was also characterized by microhardness measurement. A diamond Vickers indenter was used, applying a maximum force of 5 gf at a constant rate of 0.1 N s⁻¹ and a dwell time of 15 s.

The bond strength tests were performed according to ASTM C633 [26]. Powders were sprayed onto 5 mm thick substrate of 25.4 mm diameter. To evaluate cohesion strength within the coating, the tensile strength of as-sprayed coatings was determined by tubular coating tensile test (TCT-test). Due to the comparatively low effort in the sample preparation, this method served as an additional process control, providing information on the cohesion quality complementary to the deposition efficiency and coating microstructure. In the TCT-test, two cylindrical substrates are fixed face to face by a screwable holder, which is later fixed to a lathe chuck. The substrates remain in this fixed position during preparation and during the coating process. The specimens were pulled after unscrewing from the holder, using the same tensile machine equipment as for the bond strength test. SEM observation of the fracture surface of TCT specimens was performed to understand the failure mechanism.

3. Results and discussion

3.1. Hardening

The mean value of the in-depth measurements of micro-hardness and the standard deviation of hardness for different sets of specimen are reported in Table 3. The microhardness distribution in different series of specimen

didn't reveal any considerable differences. This clearly affirms that the change of micro-hardness by different deposition method of cold spraying was not appreciable. The indentation size is in the order of a single splat of the coating.

It has been affirmed that the width of the diffraction peak at half of the maximum (FWHM) measured by XRD is able to reflect more aspects of surface work hardening which cannot be revealed by micro-hardness values [28],[29]. FWHM appears to interest the only layer where the measurement is done. On the contrary, micro-hardness involves a finite thickness of material, and the results are an average value on the thickness of material where the indentation has been done. The FWHM is related to the grain distortion, the dislocation density and grain size. It is also assumed as an index of hardening of the material. The in-depth FWHM distribution of coated specimens is illustrated in Figure 2. It can be seen that FWHM results don't show noticeable variation in different series too.

Table 3. Average micro hardness and standard deviation for different series of specimen.

| Group name | Average microhardness (Vickers) | Standard deviation (Vickers) |
|------------|---------------------------------|------------------------------|
| CS-1P | 45.32 | 6.96 |
| CS-2P | 48.21 | 8.59 |
| CS-3P | 45.45 | 8.34 |
| CS-T2 | 43.96 | 7.16 |
| CS-T3 | 49.55 | 6.32 |

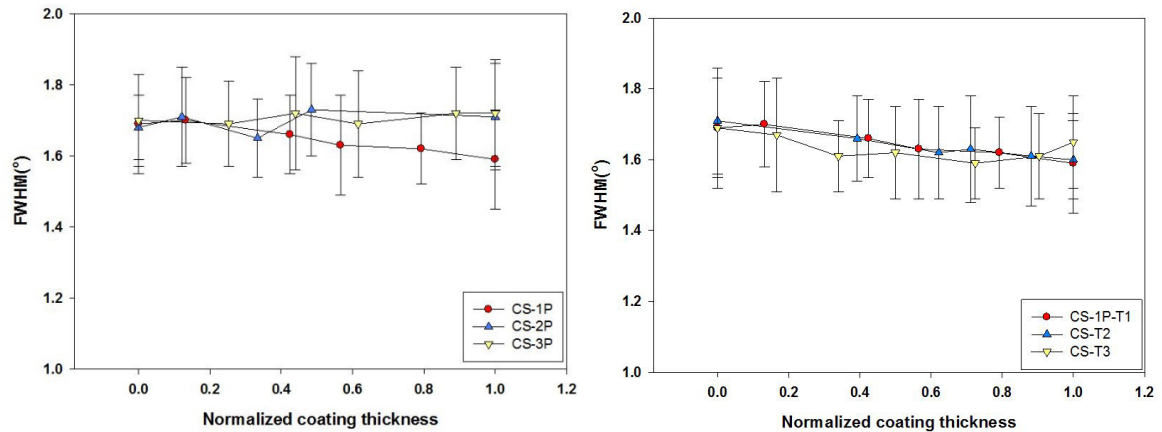


Figure 2. In depth FWHM distribution of coated specimens a) with different number of passes. b) with different coating thicknesses.

3.2. Residual stress

Residual stress buildup during cold spray coating has a great importance. Beside its effect on fatigue behavior [30], it can subsequently affect the adhesion of the coating to the substrate. In cold spray process, significant peening stresses are generated during the impact of particles on the substrate. In addition, the cold spray particles are deposited at relatively low temperatures. The net result is that cold-sprayed coating, unlike most traditional thermal spray coatings, is typically in the compressive residual stress state [22], [23]. Compressive residual stress at the interface is known to inhibit the formation of through thickness cracks and to improve bonding [31]. Since cold-sprayed coatings generally have no tensile residual stress to drive the opening or extension of cracks in the coating material, most ductile metals can be cold sprayed to almost any desired thickness.

In depth residual stress distribution of coated specimens is depicted in Figure 3. It is worth noticing that the state of residual stress in the coating is completely compressive. This clearly affirms that cold spray is not only a process that covers the surface, but also is able to generate compressive residual stress, which is beneficial in terms of fatigue behavior. Residual stress distribution of single and multiple pass deposition is depicted in Figure 3. Results

show that the residual stress has almost the same distribution in single and multiple pass deposition. Moreover, the values of residual stress at the surface and the interface between the coating and substrate are close for different number of passes. There is also no sudden drop or raise at the interface between subsequent layers because of the round shape of the specimens. In contrary, for different coating thicknesses, the residual stress at the interface is quite different. CS-T1 specimen has greater compressive residual stress comparing the other two series with thicker coatings. The subsequent effect of residual stress on bonding strength will be discussed in section 3.3.

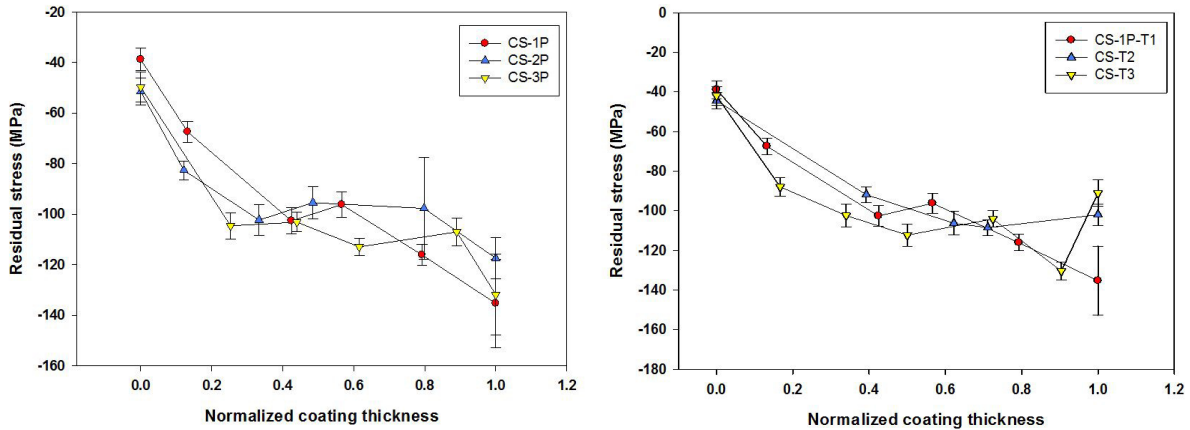


Figure 3. In depth residual stress distribution of coated specimen a) with different number of passes b) with different coating thickness.

3.3. Bond strength and tubular coating tensile test

Mechanical and physical properties of coating strongly depend on the degree of bonding between particles. The adhesion level of aluminum coatings with different deposition methods and thicknesses is evaluated through bond strength test. Five specimens were tested for each series. The average failing stress and the standard deviation are summarized in Table 4. It should be mentioned that the results of bond strength tests are not admirable, taking into account the potential capability of cold spray. For example, higher bonding strength could be achieved by heating the substrate or further increasing gas temperature or pressure. However, this study has been intentionally devoted to verify the capability of the cold spray process by implementing and using a highly reliable and long-term stable cold spray process with a well-known base and coating material. In this sense, higher gas pressure and temperature could induce rapid nozzle deterioration and as a consequence a loss in process stability and reliability. Moreover, heating of the substrate could be complex in some maintenance, repair and operation (MRO) applications, in particular for in situ applications or where large components are considered and the risk of small distortion is relatively high. Finally, it will be the specific industrial application to define the requirements and procedures to individuate the right compromise between coating repair performances and process requirements and reliability.

Table 4. Results of bond strength test.

| Group name | Bonding stress (MPa) | Standard deviation |
|------------|----------------------|--------------------|
| CS-1P | 23.2 | 2.8 |
| CS-2P | 20.7 | 4.3 |
| CS-3P | 23.9 | 4.2 |
| CS-T2 | 13.7 | 2.4 |
| CS-T3 | 13.2 | 2.7 |

The focus of bond strength test is on interface between the coating and substrate, although in some cases failures may occur partly in the coating (cohesive) which was not the case in our study. To interpret the results of bond strength tests we studied the state of residual stress at the interface. Comparison revealed that the bond strength results variation of different series is in accordance with respective residual stress variation at the interface.

As it was illustrated in section 3.2, different number of passes doesn't have a significant effect on residual stress and it can be seen that single and multi-pass series have almost the same adhesion strength. Even it can be seen that CS-2P has slightly less compressive residual stresses at interface likewise less bonding strength. Moreover, it was observed that increasing the coating thickness decreased the amount of residual stress at the interface. Similarly, it can be seen here that CS-T2 and CS-T3 series has less bond strength. This observation shows the importance of thermal input and consequently residual stress states in cold spray specimen and its effect on mechanical properties.

To evaluate cohesion strength within the coating, the tensile strength of as-sprayed coatings was determined by tubular coating tensile test (TCT-test). The results are summarized in Table 5. Stress analysis of the specimens during testing shows that the geometrical design of the two coated substrates leads to a local stress concentration in the pulled coating (notch effect) and increases the Mises stress at the gap between the substrates by a factor of 1.5. This effect must be considered for estimating real mechanical coating strength [27]. The correction factor is implied to the obtained values and the corrected results are also presented. The results have significant variation from 77 MPa to almost half of it. To interpret the results, microscopic observation of the cross section of coatings (section 3.4) in addition to SEM observation of fractured surfaces (section 3.5) are studied.

Table 5. Results of tubular coating tensile tests.

| Group name | As measured-cohesion strength (MPa) | Standard deviation | Corrected cohesion strength (MPa) |
|------------|-------------------------------------|--------------------|-----------------------------------|
| CS-1P | 77 | 1.6 | 115.5 |
| CS-2P | 39.4 | 5.9 | 59.1 |
| CS-3P | 57.7 | 5.6 | 78.6 |
| CS-T2 | 74.7 | 1.22 | 112 |
| CS-T3 | 70 | 4.75 | 105 |

3.4. Microscopic observation

Figure 4 shows the cross-section optical microscopy observation of the cold sprayed specimens. A slight variation of the coating thickness can be seen in different parts. Comparing the effect of number of passes (with the same thickness) on the microstructure of coating (i.e Figure 4a, b and c), it is obvious that the coating deposition performed by one pass of the gun has a more dense structure. In case of two and three pass deposition, there is a porous microstructure at the interface between subsequent layers. The localized increase in porosity in subsequent passes can be as attributed to the following: in multiple pass depositions, when the previous layer is formed and the subsequent pass is in progress, the infirm particle at the outer layer of previously deposited layer may detach as a result of contact of incoming particles. They may go away from the coating or they may be trapped in the structure, but they are poorly adhered. During polishing of the specimen, the particles that are weakly connected or trapped in the microstructure will detached from the coating and result in such a porous microstructure that can be seen in Figure 4 b, c). The microscopic observation of the microstructure confirms the results of cohesion tests in section 3.3 which showed less cohesion strength in multiple pass in comparison to single pass. The slight increase in cohesion strength from two pass deposition to three pass deposition can be also observed. The discussion and interpretation of this result is conducted in section 3.5 after observing fracture surfaces of broken specimen.

Increasing coating thickness doesn't have too much effect on microstructure of coatings (i.e. Figure 4 a, e and f). Nonetheless, it should be considered that these results might be valid for the investigated range of coating thickness (up to 0.8 mm). The coating density remains constant, but at the same time, the amount of voids increases by increasing the coating thickness. This results in more defects within the coating. That's why there is a slight decrease in cohesion strength by increasing the coating thickness from 500 to 800 μm .

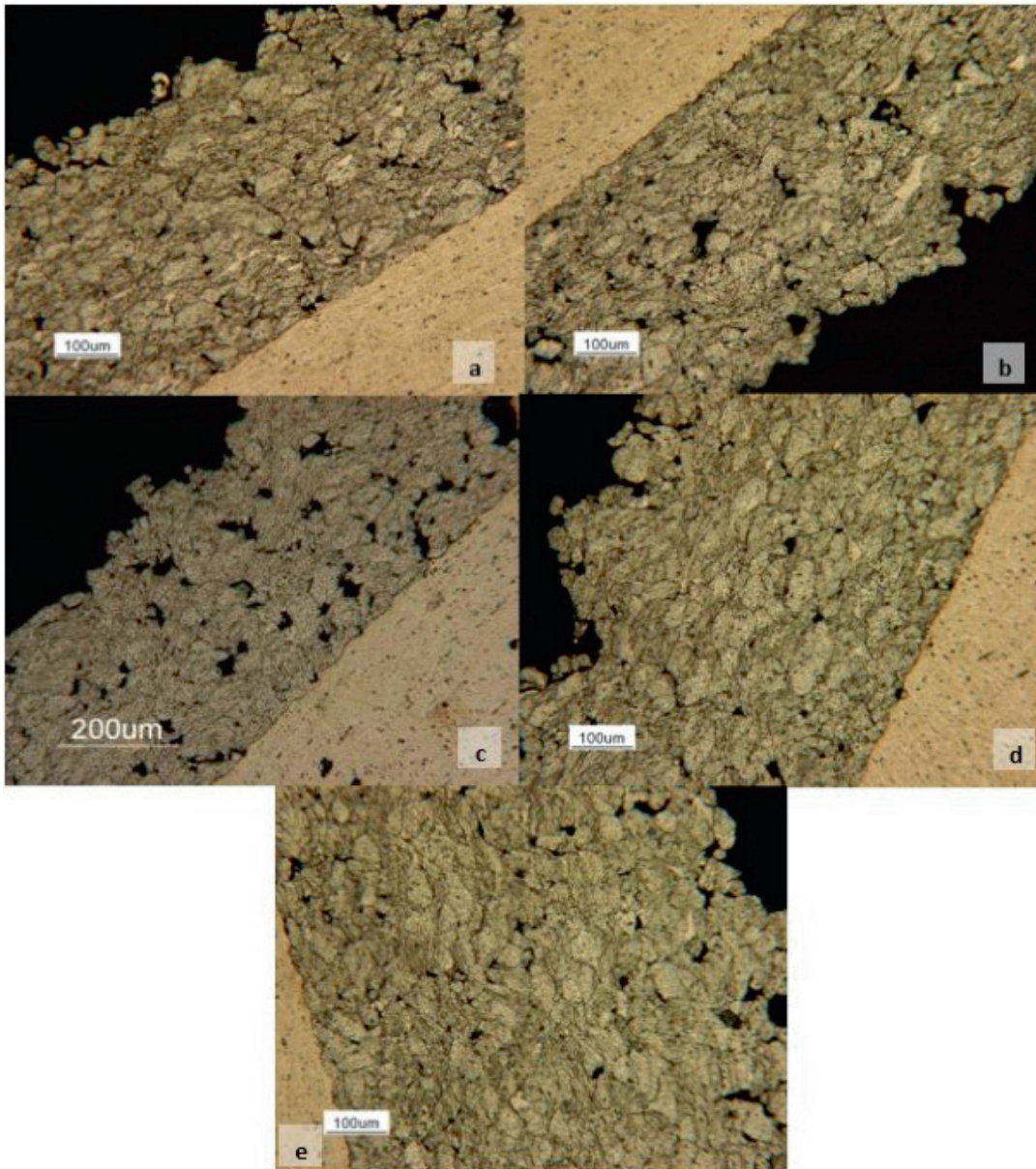


Figure 4. Cross-section optical microscopy observation of the coatings. a) CS-1P, b) CS-2P, c) CS-3P d) CS-T2 e) CS-T3

3.5. Fractography

The fracture surface of the tubular coating tensile test specimen is depicted in Figure 5 for different groups of specimen. Comparing Figure 5 a, b and c, it is obvious that by increasing the number of passes, the mixed mode fracture becomes present at the outer layer of the coating. The outer layer actually is deficient in peening effect and is generally the weakest layer.

Previous experiments indicated that the mixed - mode fracture occurs by interfacial crack propagation in layered materials. Interfacial crack propagations can be observed from Figure 5 b,c.

This is due to the fact that in the subsequent number of passes there are pores and infirm particles which play the pre crack role.

Since the coating thickness is the same for the first three groups of specimen, in three pass deposition, the outer layer is thinner than two pass deposition so the contribution of mixed mode fracture is less. Moreover the driving force for mixed mode is less than the tensile mode. This is the reason why a slight increase can be observed in cohesion strength in three pass deposition respect to two pass as it was shown in section 3.3. Comparing Figure 5 a,d and e shows that thickness of the coating doesn't have an effect on the fracture surface.

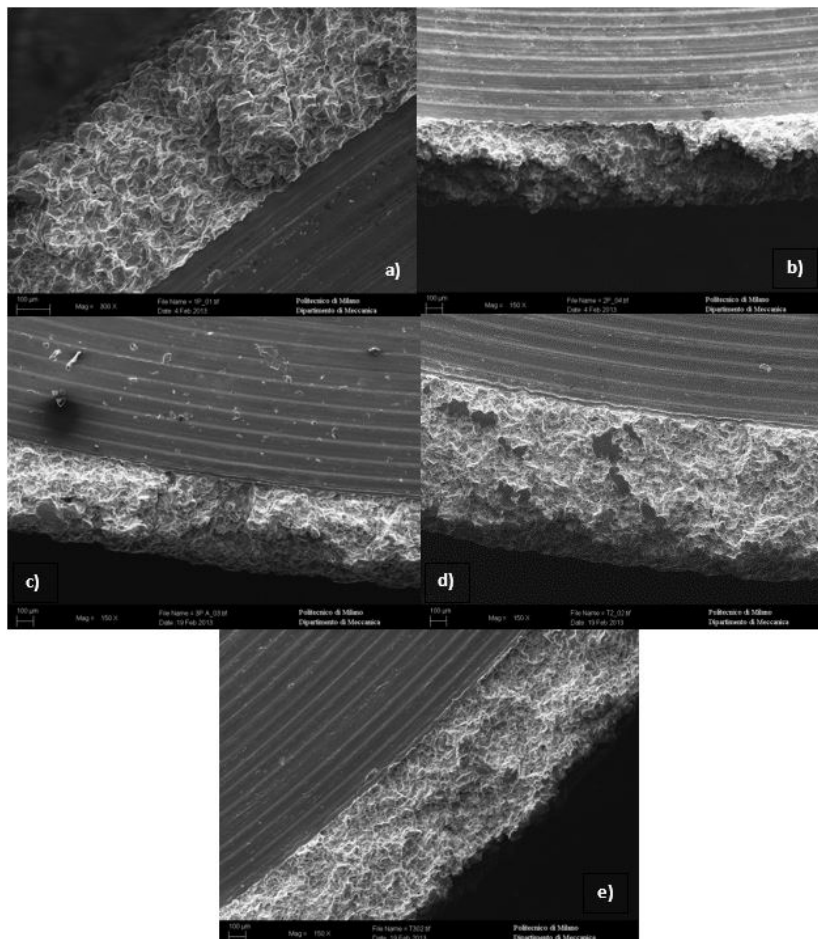


Figure 5. Fracture surface of tubular coating tensile test specimen. a) CS-1P, b) CS-2P, c) CS-3P d) CS-T2, e) CS-T3

Conclusion

The effect of number of passes in addition to the thickness of cold spray coating on cohesion and adhesion strength was studied. Cold spray conditions (gas pressure and temperature) that allow long-term process stability and reliability are employed. In particular, relatively high deposition pressure, which enhances coating cohesion and

peening effect, was used for cold spraying. The developed residual stress, adhesion and cohesion strength of the coating were examined. The following conclusions can be drawn:

- XRD results show that the cold spray process in all groups of specimen developed compressive residual stress in the coating. Number of passes had an insignificant effect on the residual stress. However, increasing coating thickness led to a progressive release of the residual stress at the interface between coating and substrate. Less compressive residual at the interface was obtained for the thickest coating.
- The bond strength results variation of different series is in accordance with respective residual stress variation at the interface. Therefore, increasing coating thickness resulted in decreasing both the compressive stress state at the interface and bond strength. Number of passes didn't have a substantial effect on bond strength of the coating.
- Microhardness measurement showed that different sets of coated specimen have almost the same hardness. FWHM obtained with XRD also didn't change significantly by changing the number of passes and thickness.
- The most important effect of different number of passes was observed in cohesion strength of the coating. In two pass deposition the cohesion strength decreased in comparison to the single deposition method. Moreover, increasing the number of passes from two to three resulted in an increase of cohesion strength. The results were interpreted by the photography of fractured surfaces.
- Fractography of specimen with different number of passes revealed that by increasing the number of passes, the mixed mode fracture becomes present in the outer layer of the coating since in a subsequent number of passes there are pores and infirm particles which act as pre cracks.
- Since the coating thickness is the same for the first three groups of specimen, in three pass deposition, the outer layer is thinner than two pass deposition so the contribution of mixed mode fracture is less. Moreover the driving force for mixed mode is less than the tensile mode. This is the reason why a slight increase can be observed in cohesion strength in three pass deposition respecting to two pass.
- Increasing coating thickness only slightly decreased the cohesion strength of the coating because there were more pores and defects in thicker coating.

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