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SURFACE and INTERFACE ANALYSIS

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Mechanical characterization of low-pressure cold-sprayed metal coatings on aluminium

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Metal coatings are widely used in order to improve the superficial properties of mechanical components or tools. Cold spray deposition is an attractive technology that allows to realize a reported coating on a metallic or polymeric surface. The essence of the phenomenon is that particles of ductile metals or alloys, having diameter of approximately 10–100 micron, become deformed and strongly attached to a surface when they impinge on the surface of metals, ceramics or glasses at impact velocities in the range of about 400–1200 m/s. In this manner, coatings can be formed on a substrate. In this work, mechanical tests were carried out in order to evaluate the adhesion of different metal particles on an aluminium alloy substrate, in order to characterize both the coating-substrate adhesion and the adhesion between two consecutive layers of the cold-sprayed metal coating. In particular, both bending tests and pure adhesion test were carried out in order to better understand the powder deposition mechanism. Bending tests were carried out in four-point bending configuration. On the basis of the experimental campaign, it results that the adhesion of the pure aluminum particles are very good, conversely the copper particles show a good adhesion on the aluminum substrate but have a low internal cohesion. **Copyright © 2013 John Wiley & Sons, Ltd.**

Keywords: cold spray; aluminum; adhesion; bending

Introduction

Cold gas dynamic spray technique is a technology of deposition that consists in the realization of surface coatings with high-velocity metal particles sprayed on the substrate at temperature significantly lower than the melting temperature of the substrate itself.

In the past, such coatings have been applied using thermal spray process. In conventional thermal spray process, the coating material is heated to molten or semi-molten state and impinged onto a substrate surface to make coatings. Consequently, thermal spray coatings have brought the problem caused by the heating process, i.e. oxidation, phase transformation, etc. In thermal spraying, bonding between splats and the closing of pores can be enhanced by high temperatures or high particle velocities during particle impact. The thermal and kinetic energy contents of particles impinging the substrate vary from one process to another. However, for metallic materials or composites, high process temperatures can increase the amount of oxides embedded in the coating and therefore reduce their performance in technical applications. In flame spraying or high-velocity oxy-fuel (HVOF) flame spraying, possible reactions of the feedstock material with the oxygen needed for combustion, with the combustion products, and with the oxygen in the air mixed into the free jet have to be considered as well as the oxidation of hot coating surfaces. In some cases, the reaction of the spray material with the ambient atmosphere can be reduced by shrouding the flame with an inert gas. Oxidation can also be limited for processes that do not require the presence of a combustion flame, like plasma spraying (PS) or arc spraying, by using a controlled atmosphere in a closed chamber. However, the comparison of atmospheric PS and vacuum PS (VPS) has already shown that the latter can be performed only at significantly higher costs. Moreover, in comparison to processes operating in the ambient atmosphere, the application of VPS does not allow sufficient cooling of the base material, which therefore has to tolerate the effects of enhanced temperatures. These include solid-state phase transformation and phase growth as well as the thermal distortion of complex components. Therefore, over the last two decades, the development of flame-based spray systems that work in ambient atmospheres has been aimed at reducing the temperature of particles and increasing their velocity. In HVOF spraying, higher particle velocities were obtained by using converging-diverging de Laval-type nozzle designs and higher gas pressures. With a chamber pressure of up to 1 MPa, HVOF spray systems of the third generation accelerate spray particles to velocities of about 650 m/s. Coating microstructures demonstrate that only small particles or fractions of larger ones are molten before the impact onto the substrate. A further reduction in particle temperatures below the melting temperatures of metals requires a substantial increase in velocity, which can only be realized by optimizing the expansion ratio in the diverging nozzle section and by using higher chamber pressures.

Following an alternative concept working without plasma or flame, the development of cold spraying started about 20 years ago by a group of scientists at the Institute of Theoretical and Applied Mechanics, Novosibirsk, Russia. This process of deposition utilizes metallic particles in order to form a coating upon a suitable substrate. The powders are accelerated by injection into a high-velocity stream of gas generated through the expansion of a pressurized gas to supersonic velocity. The idea of using a compressed gas jet to accelerate metal particles to supersonic velocity in order to produce a coating by impact of the solid particles onto a substrate was patented as early as 1903 and 1963. However, successful development of this technique and

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fabrication of commercial systems emerged only several decades later. The metal particles are accelerated and impacted onto a substrate after exiting from an appropriate gun and create a bond with the substrate. As the process continues, particles continue to impact the substrate and form new bond with deposited materials in a uniform coating with little porosity and high bond strength. This process allows to carry out several metallic coatings at relatively low temperature (substrate temperature will be lower than 100 °C). The problem of the high-temperature deposition process, as the residual stress that develops at interface causes desponding, with this technology is minimized or eliminated with the cold deposition of the metal.

Nowadays, there exist two categories of such systems, namely the high and the low-pressure guns working at typical stagnation gas pressure ranges of 2–5 MPa and 0.3–1 MPa, respectively. High-pressure systems allow achieving a higher particle velocity as compared with low-pressure systems, which in turn provide higher deposition efficiency and broader range of eligible materials. The major drawback of these systems is high operation cost as they use high-pressure gas (N_2 or He) at high flow rate (120–220 m^3/h). In low-pressure systems, metals such as Al, Zn, Cu, Co and Ni can be deposited with fairly 'good' coating properties but with low deposition efficiency. As demonstrated by the fast increasing number of contributions to the annual meetings of the International Thermal Spray Society, cold spraying nowadays is attracting serious worldwide attention. Current studies on the topic are aimed at optimizing nozzle designs, exploring possible bonding mechanisms, or demonstrating the applicability of various spray materials. For a deeper understanding of the process, the relationships between the deposition efficiencies and particle velocity were also investigated.

Cold spray is an emerging coating technology[1] that makes use of a converging/diverging nozzle and a high pressure, heated gas source (usually nitrogen) to create a high-velocity gas flow. Metallic particles, usually in the size range of 10–100 μm, are injected into this gas flow and propelled to supersonic velocities. Coating deposition occurs at relatively low temperatures compared to other spray technologies, with the sprayed particles remaining in the solid state. When processing conditions are optimized, the technique can produce nearly fully dense coatings. Despite the promise this technique has shown, questions remain about the quality of the material deposited in terms of adhesion strength of coating to substrate and cohesion strength between cold-sprayed particles that make up the coating.

Typically, the adhesion or bond strength of cold spray coatings is determined using ASTM C-633-99.[2] Other methods are similar to the standard[3] and involve the deposition of coatings onto the ends of polished cylindrical grips. One face of a cylinder is coated and then glued to a counterblock with an epoxy resin. A tensile test is conducted, and, if failure takes place at or near the coating/substrate interface, an adhesion strength is calculated from the peak force and the coated area. One drawback of this technique is that substantial quantity of material is needed to coat the cylinders. Another pitfall is that if the coating/substrate adhesion strength is high, the failure often takes place in the epoxy or at the epoxy/coating interface. In this case, the test only provides a lower limit for the adhesion strength. Sometimes, a qualitative approach is taken; particle/particle interfaces are examined by light optical microscopy or scanning electron microscopy.[4] One may also use the depth obtained in a scratch tests as an indirect measure of cohesion.[5] However, when quantitative data is sought, coatings must be deposited onto specialized test geometries that require careful machining and a substantial quantity of sprayed material. In previous papers were studied different cold-sprayed coatings involving aluminum, investigating different features of the coating and studying the coating microstructure and its wear and corrosion features.[6,7] Nowadays, many researchers are focusing their attention on the adhesion between the cold-sprayed layer and the substrate, developing new methods to enhance and evaluate this adhesion.[8,9]

Aluminum and its alloys are widely used in all the engineering fields, due to their mechanical properties and light weight; in many applications, it could be useful to realize a coating with a different metal on an aluminum component in order to improve the wear and corrosion performances of the aluminum component itself [10] (e.g. it could we very useful in aeronautics to realize a titanium coating on an aluminum component for improve the corrosion resistance and allow the coupling with carbon fibre-reinforced plastics).

Few papers in literature provide numerical values for the adhesion between an aluminum substrate and different cold-sprayed metal coatings. The aim of this study is to enhance the knowledge in this field studying the adhesion between aluminum and different metal coatings by means of experimental methods, providing numerical values in terms of maximum detachment tension and maximum bending load. Both four bending test and tensile test were carried out in order to evaluate the mechanical properties of the cold-sprayed layer; furthermore, some micrographs were taken in order to appreciate the coating microstructure.

Experimental

In this study, commercial cold spray facility DYMET403J [Obninsk Center for Powder Spraying (OCPS), Russia] was used for spraying. Since this equipment uses compressed air for the carrier gas, the air compressor is only required. Moreover, the main unit has built-in small gas heating system and heats working gas directly; hence, it has a feature of small and light weight. Round-shaped exit nozzle was used. The diameter of nozzle exit is 4.8 mm. Compressed air at 0.6 MPa was used for carrier gas.[11]

Modern DYMET equipment is compact light weighted gas dynamic spray machine with GDS spray gun, two powder feeders and control unit (Fig. 1). Control unit includes air pressure and air temperature control and powder feeders switch with powder feed rate regulators. Compressed air line has to be connected to the air inlet, and operating air pressure may be adjusted to the required value by pressure regulator. Several discrete temperature settings are intended for the quick easy choice of the optimal spraying parameters. Powder feeder switch allows quick change between two powders. GDS spray gun includes light weight air heater and supersonic nozzle.[12,13] Both round and flat nozzles may be attached to the gun. The specific modern DYMET nozzle includes replaceable nozzle insert as a changeable element eroded by powder mixture. The powder injection unit in the nozzle is designed for optimal powder distribution inside the nozzle and minimization of nozzle insert wear. The radial powder injection downstream of the nozzle throat maintained in all DYMET nozzles allows the use of open powder feeders. The GDS gun is optionally equipped by single powder button switch or by handle with the trigger for air and powder switching on. Both may be easily disjoined for the use of the gun in robotic operation. Two open powder feeders keep uniform powder supply for 15 min each and may be uploaded during the operation. Regulated powder feed rate may be maintained up to 3.5 kg/h.

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Figure 1. Dymet apparatus for low-pressure cold spray deposition.

The fabrication of low-cost portable cold spray equipment, suitable for a wide number of repair and restoration applications, was possible, thanks to the new developments[14] introduced by the OCPS, Obninsk, Russia, in the 90s.

Widespread commercial development of the cold spray technology outside Russia started only in the early 2000s. Ever since, there has been increased interest in the cold spray technology as demonstrated by the exponential growth of publications and patent applications.[15] Today, all of the cold spray methods may be categorized within three main families of processes, namely high-pressure cold spray, low-pressure cold spray and shockwave-induced spraying.

This research activity was focused on the low-pressure cold spray (CGSP-L), in which pressurized air, nitrogen or helium (5–17 bar) is heated (up to 550 °C) and forced through a converging-diverging nozzle (DeLaval nozzle) where the gas accelerates to about 600 m/s. The feedstock is introduced downstream into the divergent section of the nozzle at low pressures[16] (see Fig. 2). Subsequently, CGSPL systems can be simple, portable and relatively inexpensive to operate. Low-pressure systems are best suited for spraying ductile metals such as aluminum, copper, zinc, tin, nickel

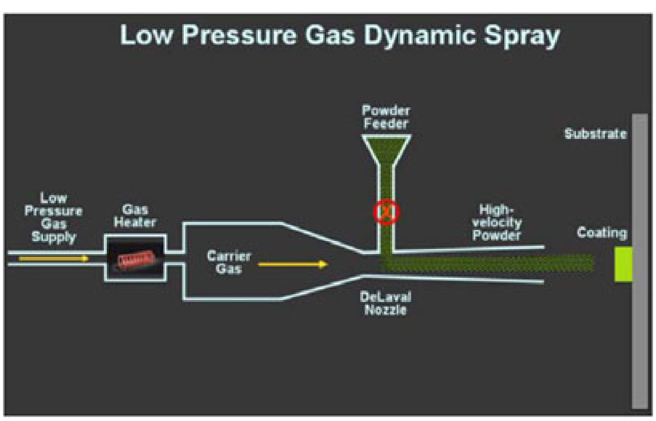

Figure 2. Low-pressure gas dynamic spray process schematization.

or even titanium onto a variety of metallic and ceramic substrates, including magnesium.

All the powders used in this experimentation were provided by DYMET, with a granulometry of about 20 microns; furthermore, all the powders were mixed with Al_2O_3 particles in order to improve the growth of the cold-sprayed layer.

The alloy AA 6060 was used as substrate, such a decision due to the wide employment of this alloy in industrial applications. Both the composition and the mechanical properties of this alloy are fully available in literature.

In Table 1 are reported the different coatings tested with the main process parameters adopted.

The process parameters were chosen after a preliminary experimental campaign that is not discussed in this paper for the sake of brevity. Some micrograph were taken using a metallographic microscope in order to appreciate the presence of defects in the cold-sprayed layer, the specimens were mounted in epoxy resin and mechanically polished.

Adhesion tests were carried out following the ASTM C633 standard, a scheme of the test configuration is reported in Fig. 3. There are two test cylinders made in AA 6060: on the former, it was deposited in the cold-sprayed layer, and in the latter, it was bonded with the former by means of araldite. The above described specimen was subjected to a tensile test, as indicated in Fig. 3, and the coating adhesion was evaluated for comparison with the araldite one. Were tested five valid specimens for each sample.

Table 1. Main process parameters of the different cold-sprayed layers

Powder sprayed	Gas used	Gas temperature [°C]	Gas pressure [bar]	Coating thickness [mm]
$Al + Al_2O_3$	Air	325°	8	0.30
$Cu + Al_2O_3$	Air	325°	8	0.30
Ti	Air	325°	8	0.05

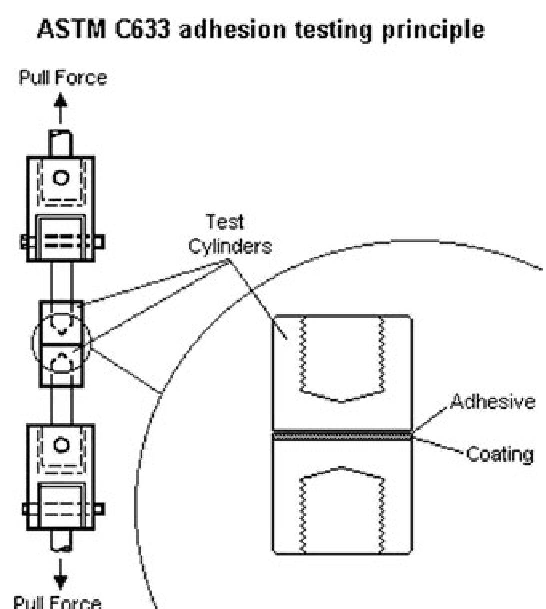

Figure 3. ASTM adhesion test principle.

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SURFACE and INTERFACE ANALYSIS

To evaluate the mechanical properties of the cold-sprayed depositions under tensile and compressive loads, four-point bending tests were carried out, following as done in previous papers.[17] Furthermore, with this test, it is possible to evaluate the adhesion between the cold-sprayed coating and the substrate under applied load. Using cold spray, 0.3 mm thick $Al + Al_2O_3$ depositions were sprayed on 3 mm thick AA 6060 substrates. By machining, the bending specimens with the size of $3 \times 13 \times 100$ mm were prepared from the cold-sprayed specimens. The samples were subjected to four-point bending tests with 40 mm inner span and 80 mm outer span using a servo-hydraulic MTS Alliance testing machine at a cross-head speed of 1 mm/min. The bending tests were interrupted several times in order to observe the coating behavior at different load values. Bending tests were carried out in both compressive and tensile conditions for the cold-sprayed layer.

Results

In Fig. 4, it is reported the cross section of the cold-sprayed layer; in particular, it is possible to see the interface between the aluminum substrate and the superficial layer. The figure is referred to the aluminum coating but is representative for all the samples. It is possible to appreciate a good adhesion between the substrate and the sprayed layer, without any void or lack of continuity. Furthermore, the whole superficial layer seems to be quite compact and uniform. As expected there is no metallurgical continuity between the coating and the substrate, and it is possible to see that the coating layers became more compact in proximity of the substrate. As expected, the substrate grain structure appears undeformed and unaffected by the spraying process, due to the low temperature achieved during the spraying process; conversely, in the thermal spray process, the high temperatures could affect the substrate microstructure. Such a result is very important especially for heat treatable alloys.[18–20] Concerning the coating, no microstructure is appreciable. It is possible to see the different splats that constitute the coating; the darker zones are due to different light reflection in dependence of the high superficial roughness. Future works have to take into account the influence of the substrate superficial roughness on the coating adhesion. Lastly, it is possible to appreciate that the coating remains compact also at high thickness, so this technology allows to realize thick coatings.

In Fig. 5 are reported the fracture surfaces of the specimens after the adhesion test. It is evident that the cold-sprayed layer has a good adhesion with the aluminum substrate in all the specimens tested. In particular, the Al and the Ti layers showed a better adhesion than the araldite: in fact, after the test, the araldite was fully detached from the cylinder; conversely, both titanium and aluminum were still adherent to the substrate. Because the titanium particles are much harder than the aluminum substrate, they penetrate the aluminum and remain firmly planted in the substrate.

Concerning the Cu coating, it was noticed a good adhesion between the cold-sprayed layer and the substrate; on the other hand, the cold-sprayed layer itself showed a low internal cohesion resulting in a disjunction of the layer: a portion remains on the substrate, and another portion remains bonded on the araldite after the tensile test. The numerical results of the adhesion test are reported in Table 2.

The aluminum and titanium coatings showed the same values of load and tension, since these values are referred at the araldite, because the araldite was detached from the substrate while the cold-sprayed layer still remains *in situ* after the tensile test. On these premises, it is possible to affirm that the reported values are the minimum possible for the cold-sprayed layer, but probably the real values are higher. Concerning the Cu coating, the values recorded are referred at the internal cohesion of the layer itself and are approximately half of the ones recorded for the aluminum and titanium coatings. On the other hand also, for the Cu coating, the adhesion between the cold-sprayed layer and the substrate was good, and the failure was due to an internal de-cohesion of the layer itself.

Concerning the four-point bending test, the load stroke curve is reported in Fig. 6, and it is possible to appreciate a typical elasto-plastic behavior. In correspondence to the red circles, the test was interrupted, and the specimen was observed in order to evaluate the behavior of the cold-sprayed layer. For the sake

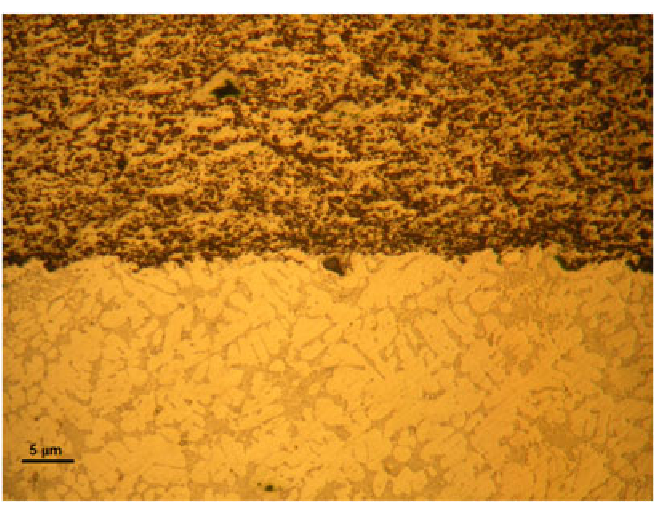

Figure 4. Cross section of the cold-sprayed layer.

Figure 5. Fracture surfaces after the adhesion test.

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Table 2. Results of the adhesion test

Sprayed layer	Maximum load	Detachment tension	Mean load	Mean detachment tension
	N	MPa	N	MPa
$Al + Al_2O_3$	15 607	31	15 591	31
$Al + Al_2O_3$	12 512	25		
$Al + Al_2O_3$	16 380	32		
$Al + Al_2O_3$	17 309	34		
$Al + Al_2O_3$	16 147	32		
$Cu + Al_2O_3$	7893	16	7862	16
$Cu + Al_2O_3$	7793	15		
$Cu + Al_2O_3$	8158	16		
$Cu + Al_2O_3$	8648	17		
$Cu + Al_2O_3$	6817	13		
Ti	15 408	30	15 208	30
Ti	16 003	32		
Ti	13 671	27		
Ti	15 256	29		
Ti	15 700	31		

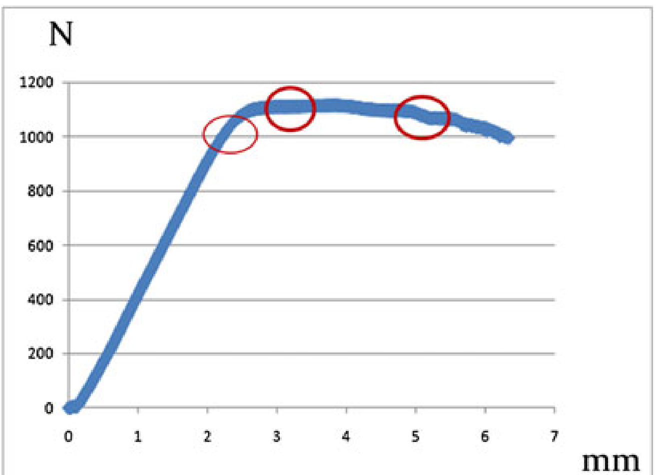

Figure 6. Stroke–load curve for the four-point bending test.

of brevity, in this paper are discussed only the results obtained for the Al coating that are representative for all the coatings tested. The macrographs taken at each step for both compressive and tensile stress are reported in Fig. 7. It is evident that, under a compressive stress, the layer remains compact and adherent to the substrate until the end of the test. On the other hand, the tensile stress is more dangerous for the coating integrity; in fact, it is possible to appreciate three different zones during the test. A first portion, coincident with the elastic field of the stroke–strain curve, the coating remains compact and perfectly adherent to the substrate. In correspondence to the yield strength, the appearance of some cracks on the coating surface is evident, and thus, it is possible to identify a second region in which the cold-sprayed layer stills remain adherent to the substrate but lost its internal cohesion resulting in the superficial cracks. Last, within the plastic region of the load–stroke curve, the coating surface appears fully cracked, and the cracks are extended in all the coating thickness. Summarizing, the coating remains

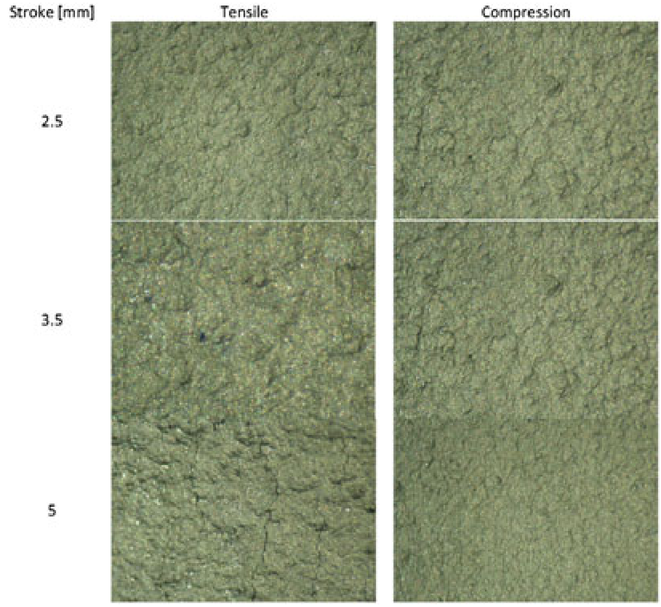

Figure 7. Surface of the cold-sprayed layer during the bending test in both tensile and compressive conditions.

adherent to the substrate for all the test duration, but, after the substrate yielding, the coating lost its internal adhesion with the arrival of some cracks. A possible explanation for this lack of cohesion can be found in the different mechanical properties of the substrate and the cold-sprayed layer. The coating behaves as a ceramic material, so it cannot follow the deformation of the substrate that behaves as a metal.

Conclusions

On the basis of the experimental campaign carried out, the following conclusions can be drawn:

- The spraying process does not affect the microstructure of the substrate base material, and the temperatures reached during the process are too low to induce any changes in the aluminum microstructure. This is an interesting result because it means that it is possible to realize a superficial coating without changing the microstructure of the substrate. This is important particularly for the heat treatable alloys.
- As expected, there is no metallurgical continuity between the coating and the substrate. This could be useful for thermal isolation and corrosion protection of the substrate. Furthermore, in the coating, no microstructure is appreciable, but the different splats that build up the coating itself are evident. The splat dimension depends on the dimension of the sprayed particles.
- All the different coatings tested were free from defects and voids, and these good characteristics are referred to the whole coating thickness. Furthermore, the interface between the coating and the substrate appears quite compact and continuous.
- All the different coatings tested showed a good internal cohesion, i.e. the coating remains quite compact during the adhesion test. Concerning the Cu coating, an internal de-cohesion of the coating was observed during the test; in fact, the failure happens in the cold-sprayed layer. Nevertheless, this failure happens for high load values, quite higher than adhesion values achieved with traditional coating methodology.

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- The Al and the Ti coatings showed a very high adhesion with the substrate resulting in a failure of the araldite bonding during the tensile test. Hence, the measured adhesion values are referred to the araldite bonding and are a lower limit of the cold-sprayed layer adhesion. Different tests needs to be carried out in order to evaluate the real adhesion between the substrate and the sprayed layer.
- The coatings showed a good adhesion to the substrate also after the bending test, but some cracks were noticed on the coating surface, indication of a lack of internal cohesion. Summarizing, the coating remains adherent to the substrate for all the test duration, but, after the substrate yielding, the coating loss its internal adhesion with the arrival of some cracks. A possible explanation for this lack of cohesion can be found in the different mechanical properties of the substrate and the cold-sprayed layer. The coating behaves as a ceramic material, so it cannot follow the deformation of the substrate that behaves as a metal.

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