Experimental characterization of thermal sprayed coatings on steel substrate

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Thermal spray technology are increasingly used in industry to create harder coatings on mechanical components to improve the wear properties or the corrosion resistance. In this paper two different coatings sprayed on a steel surface with two different techniques, high-velocity oxygen fuel (HVOF) and air plasma spray (APS), were studied. A whole experimental campaign was carried out in order to characterize the coatings and investigate about adhesion to the surface and wear properties in different conditions. Furthermore coatings with different thickness, in order to evaluate the attitude of these technologies to realize thick coatings, were studied. The results obtained suggest that both the coating types have an optimal internal cohesion, a good adhesion to the substrate and good wear resistance.

Parole chiave: HVOF - APS - Pin-on-disk - SEM - Wear - Coating

INTRODUCTION

Wear is the most common and unavoidable problem of mechanical components of cars, power generation units construction equipment, aircraft engine, chemical processing equipments. It reduces not only the life of a component but also its performance. Wear of engineering components is a considerable problem in industrial applications. To overcome this problem, wear resistant alloys or suitable wear resistant coatings deposited by thermal spray technique are generally used. Nowadays, thermal spray coatings are gaining popularity due to exceptional wear resistance property, weight reduction and cost effectiveness, as showed by Thakura1 in 2011. On the other hand there are the cold spray technology in which the coating is realized at temperatures much lower than the melting point of the particles, as studied by Astarita2 et al. in 2013.

Thermal spraying is the generic name for a family of coating processes in which a coating material is heated rapidly in a hot gaseous medium, and simultaneously projected at a high velocity onto a prepared substrate surface where it builds up to produce the desired coating. Irons3 et al. in 1996 examined the properties and cost of eight different thermal sprayed coatings and concluded that thermal spray coatings are a viable alternative to electroplated chromium in many applications. Thermal spraying is potentially a cost effective means for component dimension restoration following service induced wear. Other scientists, as Astarita4 et al. in 2013 also studied the influence of thermal treatments on the wear behavior of the materials.

In this paper, through the design of experiments methodology as done by Astarita5 et al., two different coatings sprayed on a steel substrate with different thermal spray techniques were studied. In particular, air plasma spray and high velocity oxygen fuel were studied because these are the most used techniques in industry to coat steels.

The high-velocity oxygen fuel (HVOF) thermal spray is a deposition process in which micrometric particles of metals, alloys or cermets are heated and propelled in a sonic/supersonic combusting gas stream and deposited on a substrate at high speeds to form a lamellar coating. The coatings prepared by the HVOF thermal spray process are widely used in the automotive, aerospace and chemical industries. Representative examples include WC/Co wear resistant coatings for drilling tools, YSZ thermal barrier coatings for turbine blades, and Ni-based corrosion resistant coatings for chemical reactors. The HVOF thermal spray process is characterized by very high gas and particle velocities and relatively low gas and particle temperatures, as compared to plasma spray processes. The high particle velocity helps to achieve a high particle flattening ratio at the point of impact on the substrate and to densify the coating. The short residence time in the relatively low temperature gas flame makes the powder particles highly plastic and superheating or vaporization is prevented as discussed by Mingheng6 (2006), Hazoor7 (2006) and Aw8 (2006).

Air Plasma spray (APS) is a process that combines the generation of the plasma jet, the injection and melting of particles in the plasma jet and finally the formation of the coating, it is currently the most accepted industrial practice as showed by Landes9 (2006) and Weisheng10

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(2009). Plasma spray is identified as the most flexible and versatile thermal spray process with respect to the sprayed materials. Almost any material can be used for plasma spraying on almost any type of substrate. The high temperature of plasma spray processes permits the deposition of coatings preserving the components from liquid and high temperature corrosion and wear protection and also special coatings for thermal, electrical and biomedical applications as summarized by Pfender11 in 1988 and Knotek12 in 2001. Plasma spray can deposit very high melting point materials such as super alloys and ceramics; furthermore, it offers a wide range of quality and cost effective coatings and hence can be exploited as one of the attractive means of substituting for expensive scarce materials as investigated by Ramesha13 in 2011.
Summarizing, a wide choice of coatings and coating techniques are nowadays available for industrial applications, so isn't easy to choose the more suitable one for each specific application. The aim of this paper is to characterize different coatings on a steel substrate in order to better understand the performance of each of them. Following this aim a full experimental campaign was carried out, including SEM (scanning electron microscope) observations, hardness measurements, adhesion test and pin-on-disk test.

EXPERIMENTAL

A commercial C40 carbon steel was used as substrate for all the tested coatings, in form of both plates (for SEM observations and pin-on-disk tests) and cylinders (for adhesion tests). For each coating typology specimens were realized with three different thickness, in order to evaluate the influence of the thickness on the coating performance. In particular the following coatings were tested:

- APS Cr_2O_3 99% with thicknesses of 0.05, 0.10, and 0.15 mm.
- HVOF Tungsten carbide (WC) with thicknesses of 0.04, 0.08, and 0.10 mm.

The coatings were produced using commercial equipment and taking into account the choice of the process parameters described by Gaona14 in 2008 and Fang15 in 2009. Before the thermal spray process, the steel specimen surface were sandblasted in order to enhance the adhesion. For all the coatings no bond coat was used. The process parameters adopted for each coating typology are reported in tables 1 and 2.

Primary Gas	Argon, 50 psi
Secondary Gas	He, 100 psi
Transport Gas	Argon, 30 - 35 psi
Voltage	38 - 40 V
Current	600 - 700 A

Table 1 – Process parameters for the APS coating

Tab. 1 - Parametri di processo usati per il rivestimento APS.

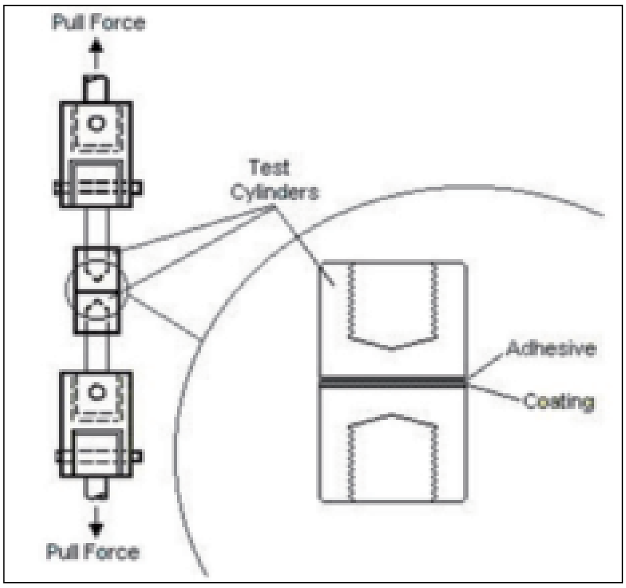

Fig. 1 – ASTM C633 adhesion testing principle

Fig. 1 - Schema del test di adesione secondo la normativa ASTM C633.

Transport Gas	Nitrogen, 0 – 6.8 bar
Oxygen	56 mc/h, 15 bar
Kerosene	7 - 11 gph
Working distance	400 - 500 mm

Table 2 – Process parameters for the HVOF coating

Tab. 2 - Parametri di processo usati per il rivestimento HVOF.

SEM observations were carried out on the top surfaces of the specimen, without any preparation. A Zeiss EVO 50 equipment was used.
Adhesion tests were carried out following the ASTM C633 standard using an universal testing machine. A scheme of the test configuration is reported in Figure 1.
There are two test cylinders made in C40 steel, on the former the sprayed layer was deposited, the latter was bonded with the former by means of adhesive. The adhesive used was araldite 420 a/b produced by Huntsman that has high performances in bonding metals. The above described specimen were subjected to a tensile test, as indicated in figure 1, and the coating adhesion was evaluated for comparison with the adhesive one. Three valid specimens for each sample were tested. The specimens dimensions were fixed following the above mentioned standard, in particular the cylinder radius was 25.4 mm with an adhesion surface of 506 mm2.
Two body wear tests were conducted on a pin-on-disk apparatus according to ASTM G99-05. In these tests, a pin slides over the coated surface of the specimens with a certain normal load, so the main test parameters are the sliding speed and the normal load of the pin. In particular the following parameters were adopted for all the tests: 5 kg normal load, 0.05 m/s sliding velocity, 30 mm

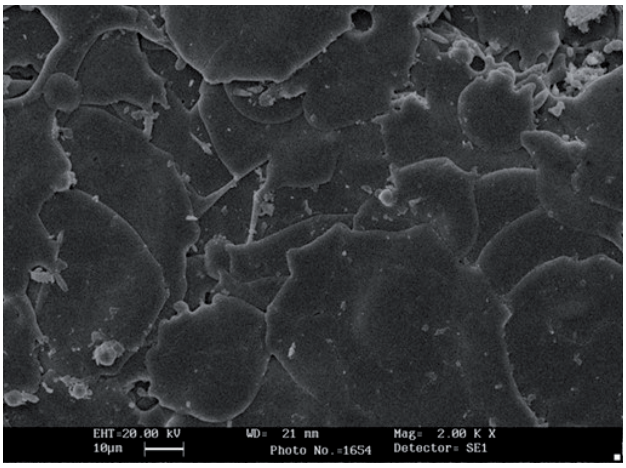

Fig. 2 – SEM micrograph taken on the top surface of the Cr_2O_3 coating

Fig. 2 – Micrografie SEM della superficie superiore del rivestimento Cr_2O_3.

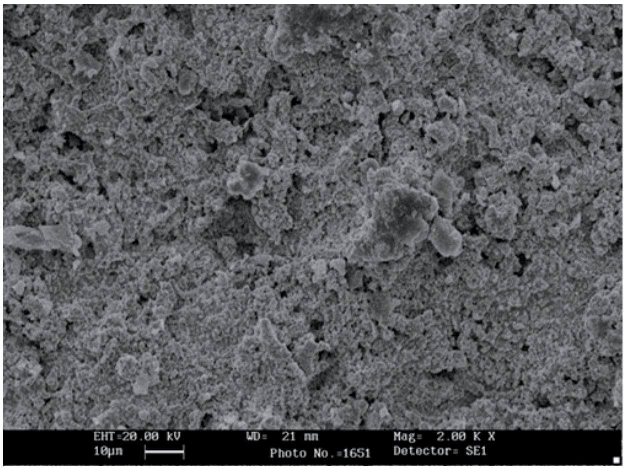

Fig. 3 – SEM micrograph taken on the top surface of the WC coating

Fig. 3 – Micrografie SEM della superficie superiore del rivestimento WC.

wear track radius and 180 m distance covered by the pin during the test. These tests were conducted using as pin a carbide insert (TPUN 220408 8016) in order to investigate the wear resistance of the coating layers in a very severe condition. Microhardness measurements were also carried out on the coated surface using a Vickers indenter with a load of 300 g.

RESULTS

The SEM image of the Cr_2O_3 coating is reported in Figure 2. The mean dimension of the splats is of about 40 micrometers and the coated surface appears quite porous. In fact it is possible to see the different layers. Furthermore the splats appear overlapped but well distinguished. A 1100 HV hardness was measured on this coating. In Figure 3 it is reported the SEM micrograph of the WC coating. In this case the splats have a mean size of 4 micrometers with a more uniform distribution with respect to the previous coating. The hardness was 950 HV but the surface appears more compact resulting in a lower porosity with respect to the Cr_2O_3 coating.
Concerning the adhesion test, the results for the two tested coatings are summarized in Tables 3 and 4 reporting the mean detachment tension and the maximum applied load as a function of the thickness. In order to better understand the phenomena occurred during the test, the images of the specimens surfaces for both coatings are reported in Figure 4. Only one specimen for each coating is shown because the behavior of one specimen is representative for all the samples having the same coating. All the samples showed a good internal cohesion of the coating. In fact for all the specimens the failure happens between the coating and the substrate or between the adhesive and the substrate, conversely the coating itself remains

Thickness [mm]	*Mean detachment stress [MPa]*	*Maximum applied load [N]*
0.05	13	6500
0.10	10	4800
0.15	8	4000

Table 3 – Adhesion test results for the Cr_2O_3 coating

Tab. 3 - Risultati dei test di adesione per il rivestimento Cr_2O_3.

Thickness [mm]	*Mean detachment stress [MPa]*	*Maximum applied load [N]*
0.04	15	8000
0.08	16	9000
0.10	16	9000

Table 4 – Adhesion test results for the WC coating

Tab. 4 - Risultati dei test di adesione per il rivestimento WC.

compact, showing a good internal cohesion resulting into the capability of the processes investigated to realize thick coatings.
Concerning the Cr_2O_3 the failure happens between the steel substrate and the coating, in fact in Figure 4 it is possible to see the filled material fully detached from the substrate, conversely the adhesive remains perfectly bonded on the surface. Furthermore, it is possible to see that the detachment stress decreases by increasing the coating thickness, and this could be a problem when a minimum thickness is required.

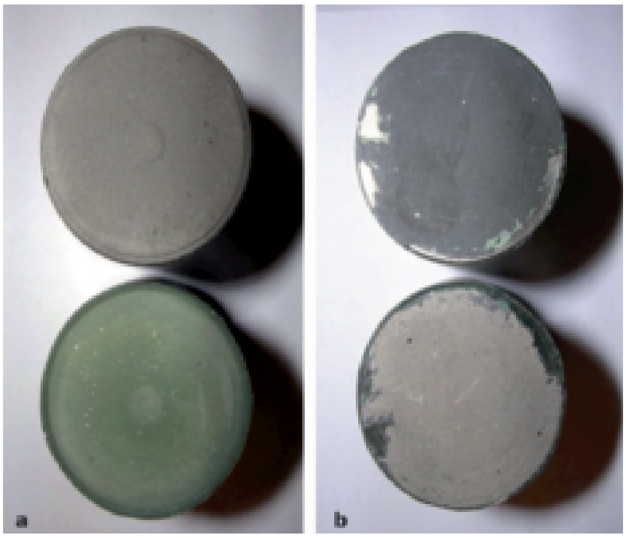

Fig. 4 – Specimens surfaces after the adhesion test: a) WC sample; b) Cr_2O_3 sample.

Fig. 4 – Superfici di rottura dei provini dopo il test di adesione: a) campione con rivestimento WC; b) campione con rivestimento Cr_2O_3

Concerning the WC coating, the failure happens between the adhesive and the substrate, so it is possible to say that the coating has a better adhesion to the substrate. Consequently, the measured forces are referred to the adhesion of the adhesive and are the same for all the different coating thickness. So the WC coating showed a good adhesion, better of the adhesive one, but with this test is impossible to appreciate the influence of the coating thickness on the adhesion due to the adhesive failure.

In the wear tests, the behavior of the two coating types was very different. In order to generate more severe wear conditions, able to damage the surface of the disks, an insert, employed as tool for chip removal, was employed as pin in the pin-on-disk tests. The insert is positioned with rake angle equal to 0 and, in order to not generate chip removal but only sliding conditions, the sliding direction is from the flank side with a relief angle of 10°, as reported in Fig. 5.

The behavior of the two coating types was different; the Cr_2O_3 coating was completely removed in the tests independently by the thickness, after 15 minutes.

For WC coating the wear, characterized by an abrasive contact, increased the loss in weight of the pin and the disk, but the weight loss increase is not linear. In Fig.6 the wear for different coating thickness is reported. It is possible to observe that the thickness has only a slight effect on the wear behaviour.

From these tests the trend of the loss weight is not perfectly linear, for this reason another test was carried out with a time test of 3 hours. In Fig.7 the results in terms of weight loss for this test are reported. It is possible to note a change in slope of the trend of wear after a time test of about 60 minutes, probably due to the generation of an extended area of contact between the insert and the disk.

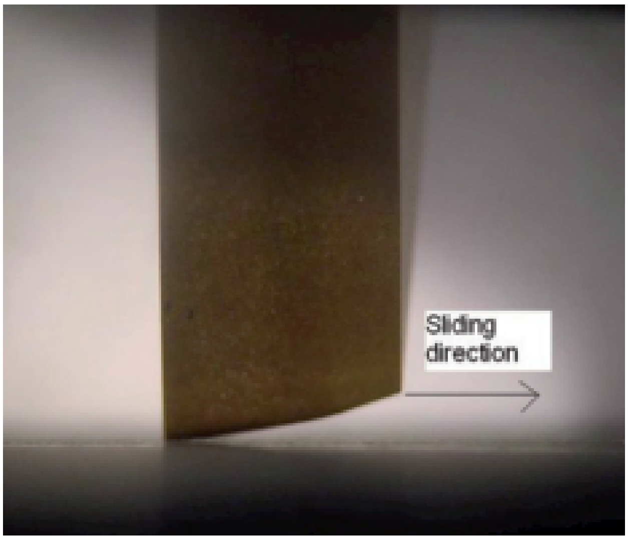

Fig. 5 – Contact disk–pin for tests with a WC insert

Fig. 5 – Particolare della zona di contatto disco-pin per le prove effettuate con l'inserto al carburo di tungsteno.

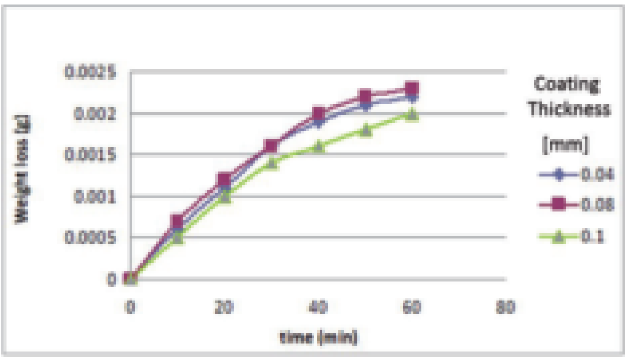

Fig. 6 – Wear for the disks with WC coating in the tests with the insert

Fig. 6 – Usura misurata sulla piastrina con rivestimento WC testate usando l'inserto al carburo di tungsteno come pin.

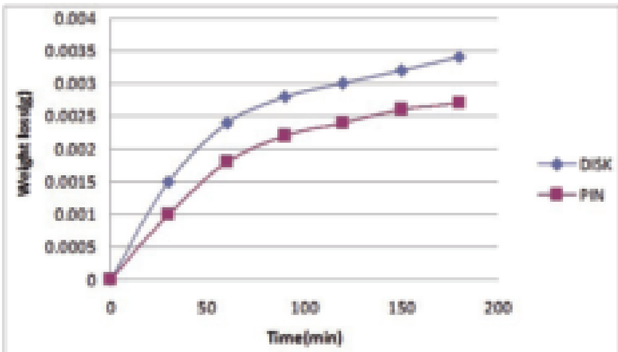

Fig. 7 – Wear for the disk with WC coating (0.1mm thick)

Fig. 7 – Usura del rivestimento in WC di spessore 0.1 mm.

Concerning the scratch test the two coatings tested showed a completely different behavior. In particular the Cr_2O_3 coating was completely removed from the substrate after few minutes, conversely the WC coating showed only a superficial roughness reduction. Such a difference is due to the different superficial morphology of the two coatings. The Cr_2O_3 coating is composed by splats with an average dimension of 40 micrometers overlayed and with a big porosity and void presence; these features encourage the material removing. On the other hand, the WC coating is composed by splats of smaller size, 4 micrometers, and is more compact so is difficult to remove some material; in fact, after an initial phase in which the coating experiences a hammering due to the pin action no material removal or other wear phenomena were observed.

CONCLUSIONS

- The HVOF technique, due to the high velocity of the sprayed softened particles, allows to obtain compact coating with low porosity and with an average splat dimension of 4 micron. The APS technique realizes less compact coatings with bigger splats (average dimension of 40 micrometers), and with a higher porosity. Both the coatings are significantly harder than the base material, with a Vickers hardness close to 1000 HV.
- Both the coatings showed a good internal cohesion, during the adhesion test the coating material remains compact and cohesive in all the specimens. Furthermore also the thicker coatings showed a good cohesion, so both the technologies are able to realize thick coatings.
- The WC coating showed an adhesion with the substrate better than the adhesive, in fact during the test the coating remained bonded on the substrate, conversely the adhesive was fully detached. On the other hand the Cr_2O_3 coating showed an adhesion inferior to the adhesive one. In fact in all the specimens the failure happens between the coating and the substrate, in particular the adhesion decreases with the coating thickness increase.
- For the extreme wear condition generated adopting an WC insert as pin, the Cr_2O_3 coating was fully removed after some minutes after the start of the test, while an abrasive mechanism yielded to a considerable wear of the disks with WC coating independent by the coating thickness.

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Caratterizzazione sperimentale di rivestimenti realizzati mediante termospruzzatura su acciaio.

Parole chiave: Acciaio - Tribologia - Rivestimenti - Prove meccaniche

Uno dei problemi più comuni e sentiti per i componenti meccanici è l'usura, in quanto non solo riduce la vita utile in servizio del componente stesso ma ne provoca un detrimento globale delle prestazioni. Per superare questo problema vengono impiegati dei rivestimenti superficiali che migliorano le proprietà tribologiche del substrato sul quale sono applicati. Una famiglia di rivestimenti superficiali molto usati per le loro caratteristiche di resistenza ad usura sono quelli realizzati spruzzando particelle sul substrato metallico da rivestire mediante processo thermal spray. Per thermal spray si intende una famiglia di processi di rivestimento in cui delle particelle sono investite da un flusso di gas caldi che le riscaldano e le accelerano facendole impattare contro la superficie da rivestire. Se determinate condizioni di velocità, temperatura ed angolo di impatto sono rispettate, le particelle non rimbalzano ma rimangono adese sulla superficie formando un rivestimento compatto. All'interno di questa famiglia di processi tecnologici esistono svariate tipologie di rivestimenti e di tecnologie di deposizione, in questo lavoro ci si concentrerà su riporti realizzati mediante processo APS (plasma spray atmosferico) e HVOF (high velocity oxygen fuel) poiché sono due tra le tecnologie più usate per rivestire acciaio. L'HVOF è un particolare processo di deposizione in cui micro-particelle di metalli, leghe o carburi, vengono riscaldate e propulse da un flusso di gas supersonico e depositate su una superficie metallica in modo da creare un rivestimento sottile di natura lamellare. Nell'APS le particelle sono inserite in un flusso di plasma caldo che le fonde e le accelera permettendone l'adesione sul substrato metallico. Benché tali tecnologie siano molto usate nella pratica industriale il lavoro da compiere per poterne comprendere appieno il funzionamento è ancora molto, soprattutto per quanto concerne le potenzialità di deposizione di materiali sempre più duri su substrati di metalli dalle elevate proprietà meccaniche, come gli acciai. In questo lavoro saranno caratterizzati riporti realizzati con queste due tecnologie usando diversi materiali per il rivestimento. I rivestimenti sono stati osservati al microscopio elettronico a scansione per osservarne la morfologia, la compattezza e l'adesione al substrato. Sono state inoltre condotte prove di usura pin-on-disk e prove di adesione secondo normativa ASTM, al fine di caratterizzare completamente le proprietà dei riporti effettuati. I risultati ottenuti hanno mostrato una buona compattezza ed adesione dei riporti e delle ottime proprietà di resistenza all'usura.