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Tribological properties of wear-resistant coatings obtained by cold gas dynamic spray

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Abstract

The aim of this study was obtaining good deposits of stellite-6 by Cold Gas Dynamic Spray (CGDS), in terms of low porosity and good adhesion and cohesion. In fact, the high strength and melting point of the investigated alloy lead to a high value of the particle critical velocity in CGDS and, therefore, good quality results are difficult to achieve. The tribological properties of the coatings were analyzed by micro-hardness measurements and pin-on-disk wear tests. Results revealed that spraying parameters can be optimized to obtain almost pore-free coatings.

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Keywords: Cold Gas Dynamic Spray; Coatings; Stellite; Tribological properties

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1. Introduction

Cold Gas Dynamic Spray (CGDS) is a recent coating deposition technology in which solid powders, with a diameter of 5-50 µm, are accelerated towards a substrate by a supersonic gas jet. Due to their high kinetic energy, particles undergo large plastic deformation during impact with the substrate. The consequent adiabatic shear instabilities

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provide material flow and heat for bonding. This mechanism actually represents the main driving force for adhesion and cohesion (Papyrin 2007, Assadi 2011, Yin 2010, Schmidt 2009). In fact, the term cold indicates that the maximum temperature reached during the process is below the melting point of the powder material. As a consequence, grain growth phase transformation and heat-affected zone are significantly reduced with respect to traditional thermal spray processes. Furthermore, an inert carrier gas is normally used, such as nitrogen or helium (Assadi 2011, Stoltenhoff 2010), which inhibits oxidation and chemical contamination. However, one of the technical issue of CGDS is the lack of coating compactness and homogeneity,-when dealing with hard coating materials, due to reduced and anisotropic plastic flow coupled with possible limited ductility. This could lead to poor adhesion with substrate and porosity formation, which significantly affect the mechanical and tribological properties of the coating. As a consequence, CGDS technique have been successfully applied in last years for deposition of ductile metals, such as copper (King 2010, Fukumoto 2009, Koivuluoto 2012, 2007) and aluminum-based alloys (Spencer 2009, Ghelichi 2012, DeForce 2006), due to their remarkable ductility. In fact, for such materials process parameters can be tuned to obtain compact coatings with good adhesion to metallic substrates. On the contrary, a few attempts have been devoted to the possible use of CGDS for hard metallic coatings (Bolelli 2010, List 2012). However, the development and spread of High-Pressure CGDS equipment provide new possibilities for using a larger selection of materials and a higher quality of the coatings. This could open interesting perspectives for the development of anti-wear and anti-corrosion applications, especially in the automotive and aerospace industries.

Materials belonging to the Cobalt based alloys family denominated stellite are widely used for their good corrosion and wear resistance properties (Riddihough 1970, Crook 1990). The presence of W or Mo and Ni, Fe, C, Si, and B concurs to increase wear and corrosion resistance, due to the formation of carbides, borides and intermetallic phases. On the other hand, the low ductility of stellite alloys makes them difficult to be deposited by CGDS. In fact, materials suitable to be sprayed by CGDS should be ductile, in order to show substantial deformations upon impact, and adhesion occurs when particles reach a critical velocity (Papyrin 2007, Assadi 2011, Schmidt 2009, 2006, Stoltenhoff 2010). Nevertheless, some authors published about the deposition of stellite coatings by CGDS, with encouraging results (Cinca 2013a).

Purpose of this work is to exploit the possibility of using CGDS as a new alternative to traditional techniques like high velocity oxygen fuel (HVOF) and plasma spray. To this aim, a preliminary study on the influence of spraying parameters on deposition behavior of stellite-6 powder was carried out, in order to find the best solutions in terms of compactness, adhesion, hardness and low porosity. The tribological properties of the surface coating were also investigated by micro hardness and pin-on-disc test.

2. Materials and experiments

2.1. Coating process

A high pressure CGDS equipment (Impact Spray System 5/11, Impact Innovation, Germany) was used in this investigation. A commercial stellite-6 powder (Diamalloy 4060NS, Oerlikon Metco, Switzerland) obtained by an atomization process, with particle size of -45+15 μm, was employed as feedstock. Coatings were deposited onto stainless steel (*AISI* 304) plates. The sample surfaces were previously degreased in acetone ultrasonic bath. The values of the main process parameters, i.e. traverse gun speed (v), temperature (T) and gas pressure (p), were identified based on literature results (Cinca 2013a, 2013b) and preliminary tests.

An experimental campaign was planned, to investigate the effect of the gas stagnation temperature and pressure on the quality of the coating. Several deposition run were carried out, varying such process parameters in the ranges 800 – 970°C and 35 – 40 bar, respectively. All the other process parameters were kept constant. In particular, stand-off distance was set to 20 mm and transverse gun speed to 300 mm/s; all the samples were coated with three passes.

2.2. Micrographic observations and hardness tests

Some of the samples were cut, embedded in resin and polished to observe their cross section by Scanning Electron Microscopy apparatus (JSM 6480L, JEOL, Japan).

Hardness measurements were carried out on coating samples deposited by using a gas temperature of 970°C and a pressure of 40 bar. A micro-hardness tester (MHT, CSM Instruments, Switzerland) with a Vickers diamond tip was used. A matrix of 100 micro-indentations was made on the top surface. Samples were previously cold mounted, moreover, with the aim of investigate the non-homogeneous mechanical properties of the coatings at the micro-scale, a low value of the applied load was chosen, in particular a normal force F_n equal to 1000mN was selected, and a sharp indenter was used.

2.3. Wear experiments

Wear properties of the coatings were measured by pin-on-disk experiments (Tribometer, CSM, Switzerland) according to ASTM G99-03. Also in this case, a gas temperature of 970°C and a pressure of 40 bar were used to coat the specimens for the tests.

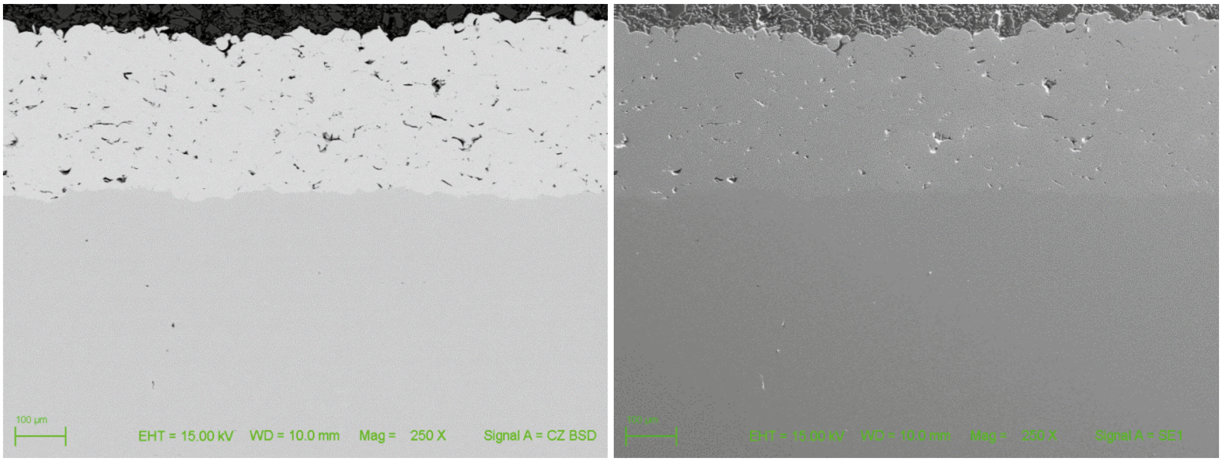
Circular samples with a diameter d=50 mm and a thickness t=5 mm were used. Before testing, the coated samples were grinded in order to obtain a flat surface (Ra=1.26±0.34 μm). The tests were carried out with a 6 mm diameter AlO_3 ball, a sliding speed of 0.06 m/s, a track radius of 12 mm, a sliding distance (L) of 1000 m and a temperature around 25° C. The friction coefficient was measured during all test under loads (P) equal to 5 N and 10 N.

The wear rate W ($mm^3\ N^{-1}\ m^{-1}$) was calculated from the volume loss in the wear track (Christian 1975), which was obtained from profilometer measurements.

3. Results and discussion

Several combinations of process parameters, in terms of gas stagnation temperature and pressure, were tested, in the ranges 800°C – 970°C and 35 – 40 bar, respectively. The quality of the coatings was evaluated on the basis of their porosity content, which is directly related to the cohesion of the deposited layer.

The better results were obtained with the set of parameters corresponding to the higher values in the ranges of the process gas temperature and pressure, i.e., 970°C and 40 bar. Fig. 1 shows the cross section of a sample deposited with such parameters. Coating was carried out by depositing three subsequent layers, resulting in a total thickness of about 320 μm.



a) b)

Fig. 1. Cross section of a sample deposited with 970°C and 40 bar, observed by SEM using (a) backscattered and (b) secondary electrons.

The reported results are consistent with the fact that the low ductility of the stellite-6 requires very high impact velocity to obtain the high deformation needed by a sound coating. Very likely, even better results in terms of low porosity coatings could be obtained by further increasing gas temperature and/or pressure. Further investigations will be carried out.

Process parameters play a very important role on the coating properties (Papyrin 2007, Assadi 2011, Schmidt 2009, 2006, Stoltenhoff 2010, Binder 2010, Arabgol 2017, Goldbaum, 2012). In fact, non-homogeneous mechanical properties of the coatings at the micro-scale are attributed to the extremely complex coating mechanisms and thermo-mechanical loading paths of each particle, starting from the gun nozzle to the impacted surface. In particular, marked plastic flows and material hardening occur during impact of the particles which represent the main driving force for adhesion mechanisms. However, non-homogenous distribution of plastic deformation could occur depending on several factors, including the local morphology and hardness of the impact zone which, in turn, are process parameter sensitive.

Local inhomogeneity was also captured from hardness measurements as shown in Figs. 2 and 3. In particular, Fig. 2 reports the Gauss probability density and cumulative functions of the Vickers micro-hardness ($HV_{0.1}$), obtained from a matrix of 10x10 indentation tests carried out on the top surface of the coated sample, together with the average value and standard deviation (μ=700.3, σ=135.0). The average value is higher than the one of stellite-6 bulk material (around 400 $HV_{0.1}$) (Koivuluoto 2007) as a consequence of the large plastic deformation and hardening occurring during particle impact. However, a significantly dispersion was obtained, with a coefficient of variation (σ/μ) around 0.2, even for the optimized samples due to the local mechanisms described above.

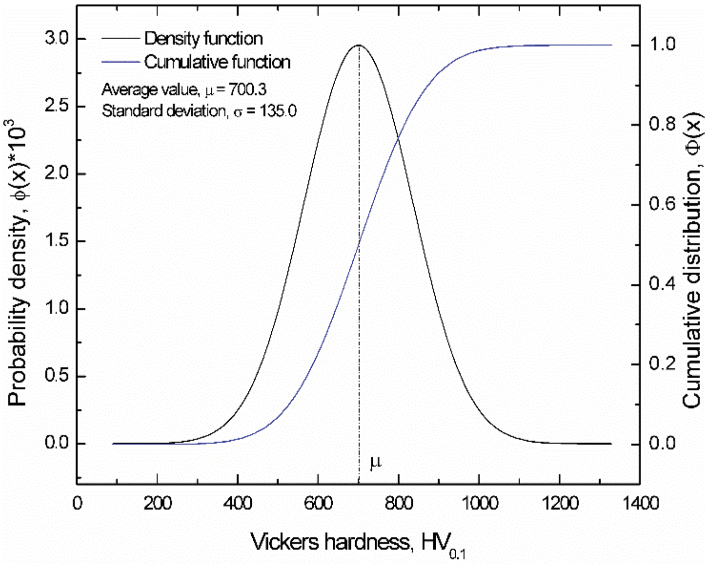
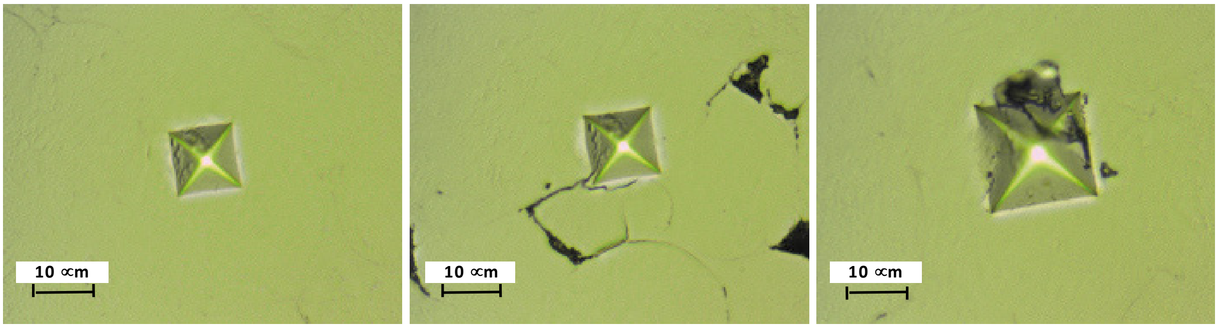


Fig. 2. Gauss probability density ($\phi(x)$) and cumulative ($\Phi(x)$) functions of the surface Vickers micro-hardness ($HV_{0.1}$) obtained from a matrix of 10x10 indentation points.



a) b) c)

Fig. 3. Microscopic observations of the indents

These local effects were also analyzed by microscopic observations of the indents as shown in Figs. 3. In particular, the figures show three typical indentations in a cracks and porosity free zone (Fig. 3.a), i.e. where one can expect that the coating mechanical properties are good, and near porous zones (Fig. 3.b and c). Fig. 3.b shows a possible debonding mechanism, induced by indentation, near the splat boundary. This observation demonstrates that splat boundaries represent a weak point of the coating. Finally, Fig. 3.c represents a typical indentation carried out near a local porosity where, as shown, the indent area is much larger and, consequently, local hardness is significantly reduced.

Figs. 4 reports the evolution of the friction coefficient measured during a wear test of a coated sample carried out under a load of 5N (Fig. 4.a) and 10N (Fig.4.b) respectively.

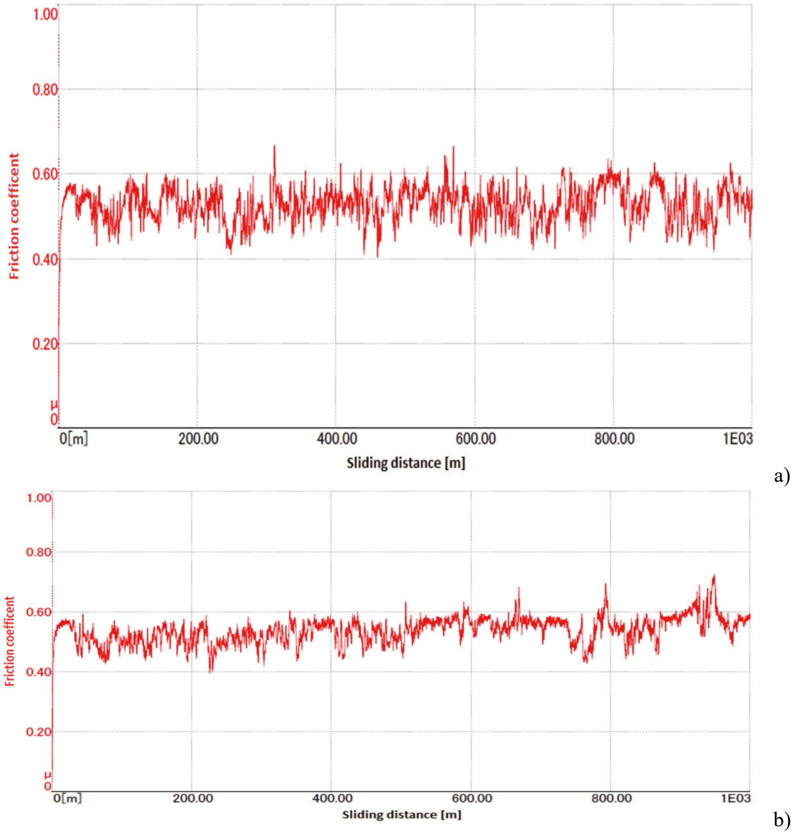


Fig. 4. Friction coefficient of two wear tests carried at 5N and 10N respectively

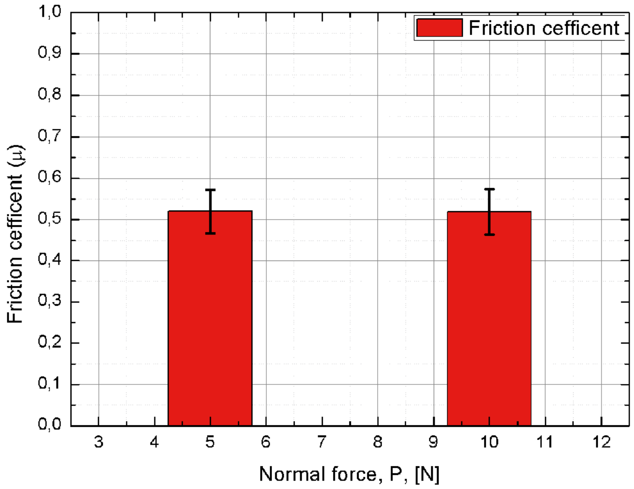


Fig. 5. Mean values of friction coefficient measured during the tests

The same behavior can be observed, as shown further in Fig. 5, in which a comparison between the two values obtained is reported. The mean value of the friction coefficient does not show significant difference between the two value of the applied load, and it could indicate that oxidation phenomena do not occur during the tests.

In Fig. 6 the results of the wear tests are reported, where it is clearly shown that the sample wear rate is higher when the applied load is equal to 5 N. This behavior could be attributed to the local damage phenomena observed also during indentation tests, as shown in Figs. 7. In particular, pull-out phenomena can be observed in Fig. 7.a, where a light micrograph of the wear track under 5 N is shown. Due to these phenomena a third-body abrasive effect can occur, which increase the surface damage. On the contrary, when the applied load is equal to 10 N, the surface of the wear track seems to be less affected by these phenomena, as shown in Fig. 7.b.

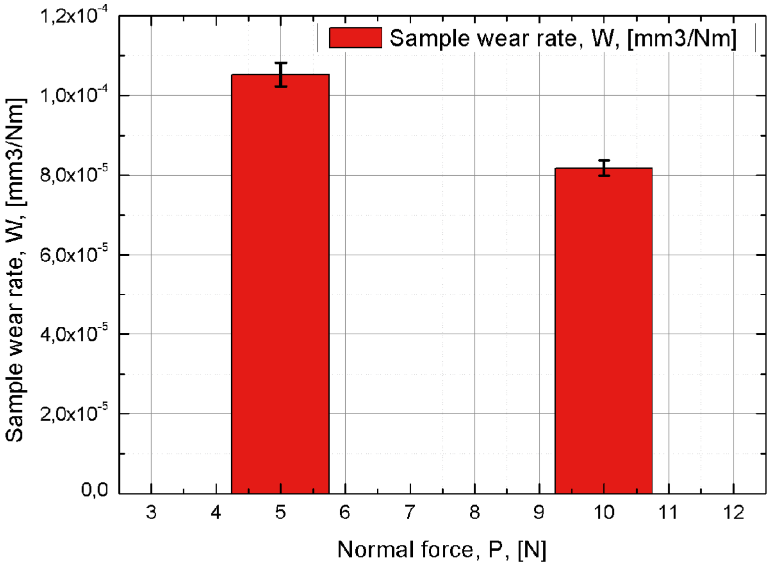
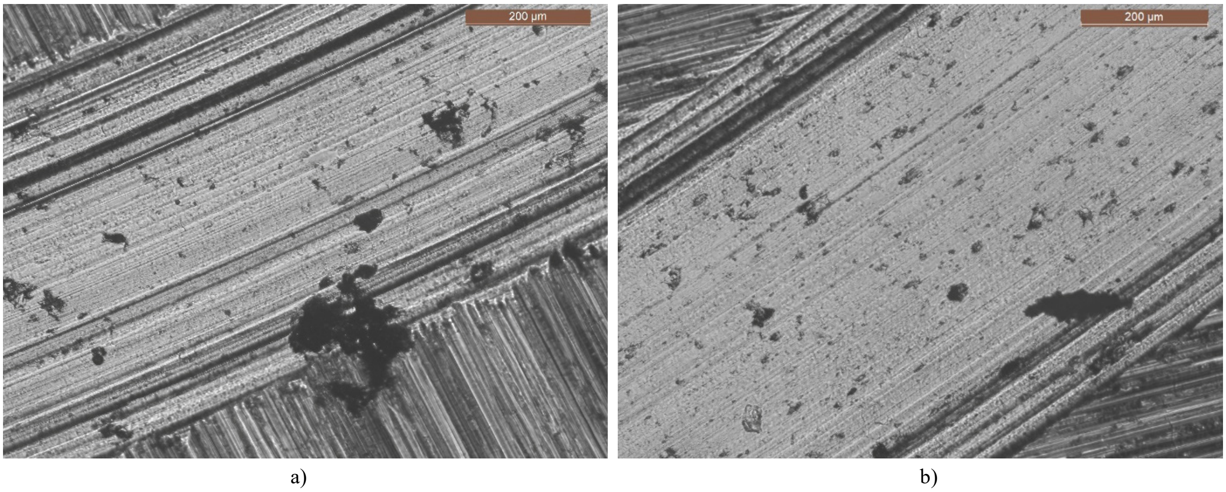


Fig. 6. Mean values of wear rate obtained from tests carried out at 5N and 10N.



a) b)

Fig. 7. Light micrograph images of the wear track for the test carried at 5N and 10N.

4. Conclusions

Porosity and oxidation on the splats boundaries are common issues in most of the coating methods whereas CGDS technique, as a solid-state process, could lead to pore- and oxide-free coatings, allowing to eliminate the dilution existing in welding processes commonly used for stellite deposition.
The main conclusions of the present work are reported as follows:

1) Cold Das Dynamic Spray has been demonstrated to be successful to produce stellite coatings. In fact, in spite of the difficulty to reach the critical velocity, good coatings, with minimal porosity and good adhesion and cohesion were obtained.
2) Good results, in terms of surface micro-hardness, were reached with a mean value of 700 $HV_{0,1}$, thanks to the large plastic deformation and hardening occurring during particle impact.
3) Hardness results show significant variability with coefficient of variation (σ/μ) around 0.2. It is attributed to local mechanisms related to non-homogeneous mechanical properties of the coatings at the micro-scale. This is confirmed by the observed debonding mechanism occurring during indentation near the splat boundary. As a consequence, splat boundaries represent weak point of the coating. Further studies will be carried out with the aim of obtaining more homogeneous properties.
4) The tribological study revealed that the friction coefficient is not affected by the applied load, in the range 5-10 N, and its low value, around 0.51, suggest that CGDS can be used in anti-wear applications. While the variation of wear rate with the applied load is attributed to local mechanisms, such as evident the pull-out phenomena observed when the applied load is equal to 5N.

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