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Deposition efficiency, mechanical properties and coating roughness in cold-sprayed titanium

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In the cold-spray process, metal powder particles develop into a coating as a result of ballistic impingment on a substrate. In cold-spray, compressed gas (air, nitrogen or helium), at pressures ranging between 1.4–3.4 MPa (200–500 psi), but typically around 1.7 MPa (250 psi), flows through a manifold system containing a gas heater and a powder feeder. The pressurized gas is heated electrically to around 100–600 °C then passed through a Laval-type converging/diverging nozzle until the gas velocities reach supersonic speeds. The powder particles are introduced into the gas stream just in front of the converging section of the nozzle and are accelerated by the expanding gas. The powder feedstock is delivered on the high-pressure side of the nozzle by the metering device, which is heated and maintained at the elevated pressure of the manifold. During the supersonic expansion through the Laval nozzle, there is a temperature reduction. Thus, the temperature of the gas stream is always below the melting point of the particulate material, providing coatings developed primarily from particles in the solid state with very little oxidation [1–5]. As cold-spray is a 100% solid-state process, the deposition "in air" of titanium coatings without significant oxidation represent an important technical achievement. Titanium and its alloys are employed in corrosive environments, aerospace and bio-implants [6].

Beyond the solid-state characteristic, a fundamental feature of the cold-spray method is the concept of critical velocity (V^*). For each coating and substrate combination there is a V^*. Above the V^* the particles will have enough kinetic energy to be incorporated into a coating. Below the V^*, the particles will be either reflected from the surface (bounced-off) or cause erosion of the substrate and any coating buildup which had begun. For particle velocities $V > V^*$, the coating process occurs and the deposition efficiency is seen to increase with increasing V [1, 4, 5].

The actual mechanisms by which the solid-state particles deform and bond has not been well characterized. It seems plausible, though it has not yet been demonstrated, that plastic deformation may disrupt thin surface films, such as oxides, and provide intimate conformal contact under high local pressure, thus permitting bonding to occur. Though unproven, this hypothesis is consistent with the fact that an wide range of ductile materials have been cold-spray deposited [4, 5]. This theory would also explain the observed minimum critical velocity necessary to achieve deposition, because sufficient kinetic energy must be available to plastically deform the solid material [1, 4, 5].

Due to this phenomenon of plastic deformation during impact, the use of a surface profilometer in order to control and evaluate the mechanical properties and deposition efficiency of cold-sprayed coatings is thought to be applicable.

The cold-spray system used in this work is located at ASB Industries, Inc. (Barberton, OH, USA) [3]. The basic scheme of the cold-spray process is described in some references [1–5] and was discussed above. During this work, the compressed gas used was nitrogen (N_2). The Ti coatings were sprayed on aluminum pipes, which were mounted and spun on a lathe. The cold-spray gun (gas heater + nozzle) was mounted on tractor coupled to the lathe, which gave transverse displacement to the gun. In order to evaluate the deposition efficiency for the different spray parameters used, all the coatings were sprayed at a constant RPM (lathe) and speed (tractor), with the same number of passes, spray time an gun displacement. Different feeding rates were used, by varying the rotation of the wheel of the mechanical powder feeder. A thermocouple was connected to the nozzle, directly on the exit of the gas heater, in order to obtain an approach of the gas temperature. The compressed carrier gas (N_2) was introduced into the powder feeder at room temperature or at 120 °C in order to attempt an enhancement of the deposition efficiency. The pressure in the powder feeder was kept slightly above the gas heater pressure in order to feed the powder particles into the nozzle. The substrates were grit-blasted with alumina just before the spraying process.

The elastic modulus of the coatings were determined via Knoop microhardness [7–9]. The arithmetic mean roughness value (R_a) of the coatings were determined by a mechanical profilometer T1000 (Hommel America Inc, New Britain, CT, USA). The relative deposition

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TABLE I The spraying parameters used for the Ti cold-sprayed coatings

Set	Gun pressure MPa (psi)	Gun temperature (°C)	RPM (hopper)	Hopper pressure MPa (psi)	Spray dist. (cm)	Pre-heating carrier gas (°C)
10a	2.07 (300)	370	1.5	2.24 (325)	1	120
10b	2.07 (300)	370	1.5	2.24 (325)	2	120
10c	2.07 (300)	370	3	2.24 (325)	1	120
10d	2.07 (300)	370	3	2.24 (325)	2	120
10e	2.07 (300)	370	5	2.24 (325)	1	120
10f	2.07 (300)	300	5	2.24 (325)	1	120
10g	2.07 (300)	300	5	2.24 (325)	2	120
10h	2.07 (300)	300	3	2.24 (325)	1	120
10I	2.07 (300)	300	3	2.24 (325)	2	120
11d	2.07 (300)	315	3	2.24 (325)	1	NA
11e	2.07 (300)	370	3	2.24 (325)	1	NA
11f	2.07 (300)	370	5	2.24 (325)	1	NA
13a	2.07 (300)	370	3	2.24 (325)	1	NA
13b	2.07 (300)	370	3	2.24 (325)	1	NA
13c	2.07 (300)	425	3	2.24 (325)	1	NA
13d	2.07 (300)	480	3	2.24 (325)	1	NA
13f	2.07 (300)	480	5	2.24 (325)	0.5	NA
13g	2.07 (300)	480	5	2.24 (325)	1	NA
14a	2.07 (300)	370	3	2.24 (325)	1	120
14b	2.07 (300)	425	3	2.24 (325)	1	120
15a	2.07 (300)	425	1.5	2.24 (325)	1	NA

efficiency was measured in the following way. The mass of the coating divided per RPM (powder hopper) times number of passes of the spray gun. The spray parameters used are listed in Table I.

According to Figs 1–3, the relative deposition efficiency, microhardness and elastic modulus of the cold-sprayed Ti coatings increase when roughness decreases. Basically the roughness tend to decrease when gun temperature increases and when spray distance decreases.

Here it is useful to think about the Madejski's equation [10]. According with Madejski's equation, the degree of spreading of a splat is directly proportional to the particle velocity and inversely proportional to its viscosity. Madejski formulated a theoretical model on the impact of a molten droplet with a solid substrate by making a relationship between the splat diameter (D) and the diameter of the initial droplet (d).

$$D/d = 1.2941(\rho \times v_d \times d/\mu)^{0.2}$$
$$= 1.2941(\mathrm{Re})^{0.2} \quad \text{(Equation 1—Madejski) [10]}$$

In this equation ρ, μ and v_d are the density, viscosity and the particle impact velocity, respectively. The Reynolds number is represented as Re. According to Equation 1, when the velocity of the particles is increased and the viscosity is decreased then particle spreading tends to increase and vice-versa.

But instead of relating particle spreading with velocity and viscosity, as in cold-spray there is no particle melting just critical velocity and plastic deformation, the particle spreading may be related to its velocity and yield stress. So it might be thought that the degree of spreading (D/d) will be directly proportional to the velocity and inversely proportional to the yield stress (σ_y) of the particle.

$$(D/d)\alpha(\rho, v_d, d)\,\alpha^{-1}(\sigma_y)$$

(Equation 2—Madejski's equation modified)

In this equation ρ, σ_y and v_d are the density, yield stress and the particle impact velocity, respectively.

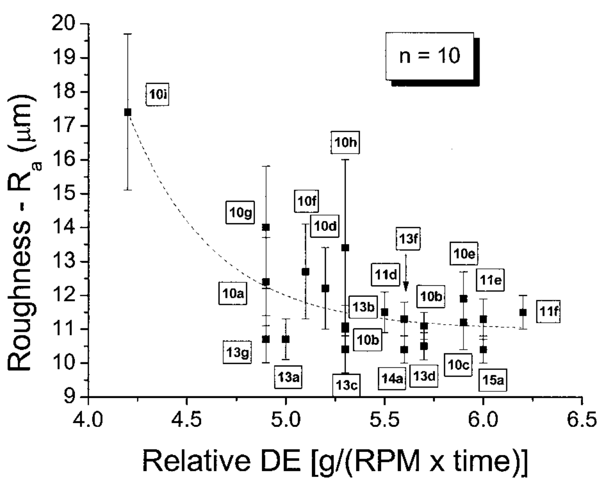

Figure 1 Roughness-deposition efficiency relationship for different sets of spray parameters for the cold-sprayed Ti coatings (see Table I).

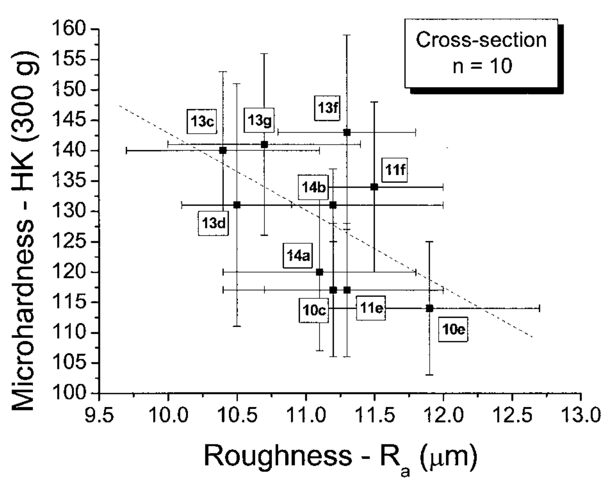

Figure 2 Microhardness-roughness relationship for different sets of spray parameters for the cold-sprayed Ti coatings (see Table I).

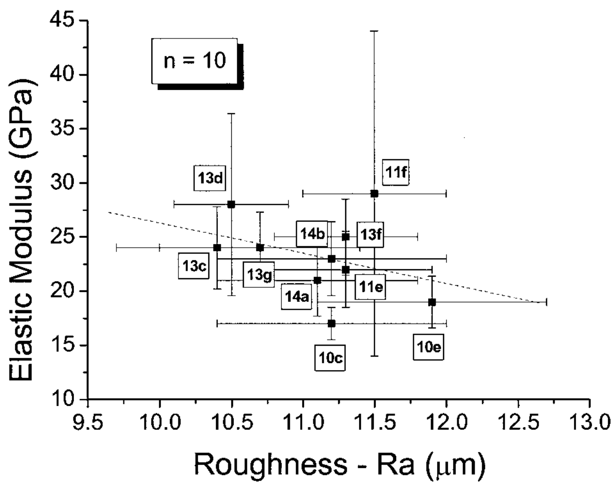

Figure 3 Elastic modulus-roughness relationship for different sets of spray parameters for the cold-sprayed Ti coatings (see Table I).

According to Equation 2, particles with the same yield stress, density and approximately same size will present larger spreading for higher velocities of impact. The low yield strength may be one of the reasons why materials such as aluminum and copper present such high density and low porosity when sprayed by cold-spray [1].

Now making a relationship between the statement above with coating roughness it seems that smoother coatings will be the result of higher impact velocities. As a consequent result, the deposition efficiency and properties related to a better packing and contact between splats, like microhardness and elastic modulus, will be improved.

Analyzing more carefully Figs 1 to 3 and the spray parameters (Table I), it is possible to observe interesting results. The lowest results of deposition efficiency and the highest results of roughness were for the samples sprayed with the lowest gun temperature and longest spray distance. When the spray distance was shortened and the gun temperature was increased the deposition efficiency increased but it reached a sort of plateau between gun temperatures of 370 to 480 °C, where the deposition efficiencies had approximately the same values for this range of temperatures.

The highest values of microhardness and elastic modulus (and the lowest values of roughness) were found for temperatures at 480 °C at 1 cm of spray distance. The results were opposite at 370 °C at the same spray distance. It should be noted here that the samples sprayed at the lower temperatures (300 and 315 °C) and longest spray distances (2 cm) were not tested for microhardness and elastic modulus. According with reference [11], the deposition efficiency is improved for higher particle velocities. According with reference [4], the velocity of the gas at the throat (V_t) of the Laval nozzle is a function of its temperature.

$$V_t = (\gamma R T_t)^{0.5} \quad \text{(Equation 3) [4]}$$

In this equation, γ is the ratio of gas specific heats (for monoatomic gases it is 1.66 and for diatomic gases it is 1.4), R is the specific gas constant (the universal gas constant divided by the gas molecular weight) and T_t is the gas temperature at the throat, respectively [4]. Equation 3 agrees with the overall results of deposition efficiency and with the more specific results of microhardness and elastic modulus.

The non-noticeable difference presented by the deposition efficiency at temperatures of 370 to 480 °C may be explained by the fact that at these temperatures the majority of the particles are able to achieve the critical velocity (V^*). The majority of the particles will adhere on the substrate or previously deposited layers. But at the highest temperatures (425 and 480 °C) the particles will also have the highest velocities of impact, having a high degree of flattening (low roughness) establishing many points of contact between splats. The enhancement of points of contact between splats will lead to higher cohesion meaning higher microhardness and elastic modulus. These results also agree with Equation 2, the modified version of the Madejski's equation.

It seems that the gas velocity is a function only of the total gas temperature. Apparently the gas pressure does not affect the gas velocity. However, it must be noticed that at higher temperatures the gas density and viscosity will decrease. As a consequence the drag force of the gas, which is the force responsible for particle acceleration should decrease at higher gas temperatures. Two important factors were noted. The pre-heating of the carrier gas and the feed rate (RPM—powder hopper) did not create any noticeable effect on the properties here analyzed. But these effects need further investigations.

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