



# Corrosion resistance of cold-sprayed Ta coatings in very aggressive conditions

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## ARTICLE INFO

Available online 23 February 2010

### Keywords:

Cold spraying  
Inert-plasma spraying  
Tantalum  
Denseness  
Corrosion properties  
Structures

## ABSTRACT

This study deals with the corrosion behaviour of high-pressure cold-sprayed Ta coatings, compared with Ta bulk material and inert-atmosphere plasma-sprayed Ta coatings. Electrochemical polarization and electrochemical impedance spectroscopy measurements are carried out in 1 M KOH solution; immersion tests are performed in 1 wt.% HF solution, and the resistance to high-temperature oxidation and molten salt attack is evaluated by hot corrosion tests. Moreover, the effect of substrate material (steel, Al and Cu) on the coating formation is investigated using FESEM.

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## 1. Introduction

Cold spraying is a solid-state process where the coating is formed by powder particles impacting with high kinetic energy, on account of their high velocity [1]. The high plastic deformation of the impacting particles produces perfect interfaces and hence highly dense microstructures [2], with no oxidation or phase transformation. Cold spraying is an optimal technique to produce dense metallic coatings for corrosion protection [1,3]. Denseness (impermeability) of the coating is indeed required when corrosion resistance is provided by a coating nobler than its substrate (e.g.: Ta on steel, Al or Cu).

Tantalum is a refractory metal ( $T_m = 2996$  °C) with excellent machinability and low ductile-to-brittle transition temperature [4]. Because of its excellent resistance against corrosion by various acids (except HF), salt solutions and organic chemicals even at elevated temperatures [5], ensured by the formation of a stable passive oxide film ( $Ta_2O_5$ ) [5], it can provide protection against extreme corrosion [6]. Tantalum coatings have been reportedly produced by plasma spraying [7], kinetic spraying [6], electrodeposition [8] and PVD techniques [9]. However, plasma-sprayed coatings are not sufficiently dense and require post-treatments (sealing or fusing) [7]. On the contrary, previous tests revealed the fully dense structure of cold-sprayed Ta, whose corrosion resistance is similar to that of bulk Ta in 3.5% NaCl and 40%  $H_2SO_4$  at 22 °C and 80 °C [10].

As cold-sprayed Ta performed well in milder corrosion conditions, this study focuses on its corrosion properties in more aggressive environments (1 M KOH, 1 wt.% HF and high-temperature molten salt

attack). Moreover, the effect of substrate material on the denseness and corrosion properties of cold-sprayed Ta coatings is investigated. Inert-plasma-sprayed Ta coatings and Ta bulk material are studied as references.

## 2. Experimental techniques

### 2.1. Coating manufacturing methods

Cold-sprayed (CS) Ta coatings were prepared at Linde AG Linde Gas Division (Unterschleissheim, Germany) with Kinetiks 4000 (Cold Gas Technology GmbH, Ampfing, Germany) high-pressure cold spray equipment, using the Amperit 151.065 powder (H.C. Starck, particle size  $-30 + 10$   $\mu m$ ). The process gas was  $N_2$  (flow rate: 82  $m^3/h$ ) with inlet pressure of 38 bar, preheated to 800 °C. The torch traverse speed was 20 m/min, the beam distance 1.5 mm, the stand-off distance 40 mm. The coatings were sprayed onto polished Al (sample label: CS Ta1) and Cu (label: CS Ta2) plates, cleaned with ethanol, and onto Fe52 steel plates (label: CS Ta3), grit-blasted with 18 mesh  $Al_2O_3$ . The coating thickness was 300  $\mu m$ .

A 1 mm-thick bulk Ta sheet (code Ta bulk) supplied by Harald Pihl Ab, Sweden (ASTM B708-98 R05200 annealed), was employed as reference. Moreover, inert-plasma-sprayed (IPS) coatings were deposited onto grit-blasted Fe52 steel substrates at Centro Sviluppo Materiali (Rome, Italy), using a Sulzer-Metco F4-VB torch in an Ar-filled chamber (parameters in Table 1).

### 2.2. Corrosion tests

Electrochemical corrosion tests were performed in contact with ~300 ml of 1 M KOH aqueous solution at room temperature in equilibrium with the environment. The tests were conducted in a 3-

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**Table 1**  
IPS parameters.

Sample label	IPS Ta1	IPS Ta2	IPS Ta3
Powder	Infralloy S7301 (Inframat Advanced Materials LLC, Manchester, CT, USA): particle size range $-120+10\ \mu\text{m}$		
Nozzle $\varnothing$	6 mm	6 mm	7 mm
Chamber pressure	919 mbar	750 mbar	300 mbar
Ar flow rate	55	55	55
H <sub>2</sub> flow rate	16	16	15
Current	650 A	680 A	680 A
Power	48.1 kW	49.6 kW	51 kW
Carrier gas flow rate	Ar: 2.2 Sl/min	Ar: 2.0 Sl/min	Ar: 2.0 Sl/min
Pass spacing	4 mm	4 mm	4 mm
No. of cycles	35	35	20
Traverse speed	400 mm/s	400 mm/s	400 mm/s
Stand-off distance	100 mm	130 mm	140 mm

electrode cell (Flat Cell K0235, Princeton Applied Research, Oak Ridge, Tennessee, USA): the sample, with an exposed surface of 1 cm<sup>2</sup>, was the working electrode, a Pt grid was the counter-electrode and the reference electrode was Ag/AgCl/KCl<sub>(sat.)</sub>. An Ametek VersaStat3 potentiostat-galvanostat (Princeton Applied Research) was employed. Cathodic and anodic electrochemical polarization tests were performed by scanning a  $[-300\ \text{mV}; +1400\ \text{mV}]$  overpotential range (vs. open circuit potential – OCP) at a rate of 0.5 mV/s. The tests were started after 50 min of free exposure to the electrolyte. The corrosion potential  $E_{\text{corr}}$  and corrosion current density  $I_{\text{corr}}$  were determined by Tafel's analysis. Electrochemical impedance spectra (EIS) measurements were acquired by applying voltage perturbation amplitude of  $\pm 50\ \text{mV}$  (vs. OCP) over a frequency range of 100 kHz–10 mHz (7 points/decade). The tests were commenced after 30 min of exposure to the electrolyte. The spectra were fitted using the Frequency Response Analyser software (Eco Chemie B.V., Utrecht, The Netherlands).

Immersion tests in 1 wt.% HF aqueous solution were carried out on CS Ta coatings (separated from their substrates) and on bulk Ta. The samples were immersed for 80 days in 150 ml of test solution at 22 °C. The CS Ta coating sample consisted of two pieces with dimensions 51 × 5 × 0.3 mm and 35 × 6 × 0.3 mm whereas dimensions of Ta bulk sample were 18 × 28 × 1 mm.

Hot corrosion/oxidation tests were performed on the CSTa3 sample and on the IPS Ta coatings. An aqueous solution consisting of saturated Na<sub>2</sub>SO<sub>4</sub> + 10 wt.% NaCl was sprayed onto the surface of 24 × 24 mm-wide coated samples ( $\approx 10\ \text{mg}/\text{cm}^2$ ), which were then inserted into an electric furnace at 705 °C. The test lasted 24 h; the samples were then allowed to cool slowly inside the furnace.

### 2.3. Microstructural characterisation

The microstructures of the CS Ta coatings were analyzed on polished cross-sections using a field-emission scanning electron microscope (FESEM, Zeiss ULTRAPLUS) whereas the IPS Ta coatings were analyzed using a scanning electron microscope (SEM, FEI XL30). The polished and etched (with nital 4%) cross-sections of coated samples before and after hot corrosion/oxidation tests were also observed by optical microscope (OM).

## 3. Results and discussion

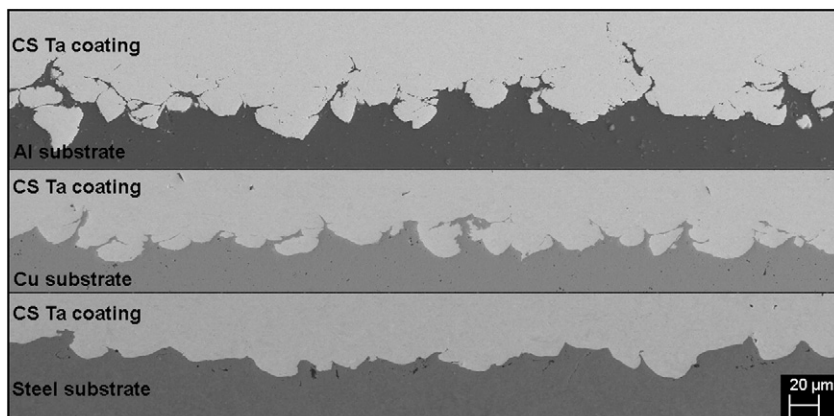
### 3.1. Microstructures of the CS and IPS Ta coatings

The interfaces between the CS Ta coatings and different substrates (Fig. 1) are voidless, indicating tight adhesion. The nature of the interfaces depends on the hardness difference between coating and substrate material. Vickers hardnesses HV<sub>0.3</sub> of the coatings and substrates are: for CS Ta on Al 239 ± 6 (coating) and 72 ± 4 (substrate); for CS Ta on Cu: 244 ± 7 and 87 ± 3; for CS Ta on steel: 243 ± 6 and 123 ± 11. The deformation level of the substrate upon particle impact is larger for softer substrates and therefore decreases in the order Al > Cu > steel. Larger embedment of Ta particles therefore occurs in the softer substrates. The hardness of the CS Ta coatings, by contrast, is not dependent on the substrate material. The Ta particles are highly deformed during impact (Fig. 2) and experience work-hardening [11]; indeed, the coatings are much harder than bulk (annealed) Ta (HV<sub>0.3</sub> = 101 ± 2).

The CS coatings have dense microstructures, with metal–metal inter-particle bonding, as revealed by Fig. 2. By contrast, IPS Ta coatings exhibit some porosity and oxide inclusions (Fig. 3). Oxides are seen particularly in the coatings deposited at low chamber pressure (IPS Ta2 and IPS Ta3), which might be due to some air infiltrations or incomplete air evacuation from the chamber.

### 3.2. Polarization behaviour in 1 M KOH

The various CS Ta-coated samples and the bulk Ta exhibit almost identical polarization curves in 1 M KOH solutions, characterised by very low current density values (Fig. 4). Accordingly, their  $E_{\text{corr}}$  and  $I_{\text{corr}}$  values do not differ significantly (Table 2). A passive stage with constant current density  $i_p = 4\text{--}5 \times 10^{-5}\ \text{A}/\text{cm}^2$  occurs in the [50 mV; 500 mV] potential range, followed by transpassivation. During reverse anodic polarization, the current density takes on lower values than during the forward scan, which indicates effective re-passivation. This



**Fig. 1.** Interfaces between CS Ta coating on Al, Cu, and steel substrates. Steel substrate was grit-blasted prior spraying whereas Al and Cu substrates were just cleaned with ethanol before spraying. FESEM micrographs.

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