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The effects of microstructural features on the performance gap in corrosion resistance between bulk and HVOF sprayed Inconel 625

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ABSTRACT

It is commonly observed that there is a performance gap between the corrosion resistance of thermally sprayed coatings and the equivalent bulk material. This is attributed to the significantly modified microstructure of the sprayed coatings. However, currently there is no detailed understanding of which aspects of microstructural modification are primarily responsible for this performance gap. In this work several deliberately microstructurally modified versions of the Ni-based superalloy Inconel 625 were produced. These were subjected to potentiodynamic electrochemical testing in 0.5 M H_2SO_4 to investigate the links between specific microstructural features and electrochemical behaviour. Samples were prepared by high-velocity oxy-fuel (HVOF) thermal spraying, laser surface remelting using a high power diode laser and conventional powder sintering. Microstructural features were examined by optical and scanning electron microscopy and X-ray diffraction. Potentiodynamic testing was carried out on the following forms of Inconel 625: wrought sheet; HVOF sprayed coatings; sintered powder compacts; laser melted wrought sheet and HVOF sprayed coatings. Using the corrosion behaviour, i.e. passive current density, of the wrought sheet as a baseline, the performance of different forms of Inconel 625 was compared. It is found that a fine dendritic structure (with associated microsegregation) produced by laser remelting wrought sheet has no significant effect on corrosion performance. Up to 12% porosity in sintered powder samples increases the passive current density by a factor of only around 2. As observed previously, the passive current density of HVOF sprayed coatings is 20–40 times greater. However, HVOF coatings subjected to laser surface remelting are found to have a passive current density close to that of wrought material. It is concluded that, whilst porosity in coatings produces some decrease in corrosion resistance, the main contributing factor is the galvanic corrosion of localised Cr-depleted regions which are associated with oxide inclusions within HVOF sprayed samples.

1. Introduction

Inconel 625 has long been used in aqueous corrosive environments due to its excellent overall corrosion resistance [1]. According to Neville and Hodgkiess [2], the passive film that forms on Inconel 625 is a mixed Fe–Cr oxide; and protects the material from further corrosion. The ability of wrought Inconel 625 to exhibit passivation at remarkably low current density values during polarisation tests is well established [3,4]. However, this is not true of thermally sprayed coatings of Inconel 625; even when deposited by the relatively new high-velocity oxy-fuel (HVOF) technique which tends to produce higher quality coatings with less oxidation and porosity than other thermal spray methods [5]. Zhang et al. [3] studied the aqueous corrosion behaviour of HVOF Inconel 625 coatings, and found that the passive current density of the sprayed coating in 0.5 M H_2SO_4 was at least five times higher than that of wrought Inconel 625. Shrestha and Sturgeon [6] remarked that thermally sprayed Inconel 625 coatings could, at best, match the performance of wrought stainless steel.

This difference in corrosion performance is generally attributed to the inhomogeneous coating structure primarily caused by pores and oxides formed during coating deposition, as well as the elemental segregation arising from the rapidly solidified structure. However, there are contrasting views in the literature about the effects of these microstructural modifications on the corrosion behaviour. For example, Shrestha and Sturgeon [6] found a more highly oxidised Inconel 625 coating to possess poorer corrosion resistance; whereas Zhang et al. [3] noted that Inconel 625 coatings with the lowest oxide content did not give the lowest current density values. The contribution of porosity is also contentious; according to Neville et al. [4], it is no longer a major corrosion issue for the HVOF process because porosity levels are typically less than 1–2% in coatings deposited under optimised conditions. Due to the nature of thermal spray processes porosity and oxidation tend to occur immediately

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Table 1
Chemical composition of Inconel 625, weight percent.

	Ni	Cr	Mo	Nb	Fe	C	Mn	Si	Mg	Al	Ti	Co
Wrought	62.9	21.8	8.9	3.42	2.44	0.01	0.05	0.07	0.01	0.1	0.2	0.1
Powder	65.1	22.0	9.0	3.43	0.01	0.01	0.1	0.2	–	–	–	0.15

adjacent to each other [7], making it difficult to determine the independent effect of each on corrosion performance.

In an effort to further improve the corrosion behaviour of coatings, a number of researchers have investigated the use of laser surface melting (LSM) [8–11]. LSM of thermally sprayed coatings has been shown to be able to significantly improve corrosion performance. This improvement is attributed to elimination of interconnected porosity as well as a general homogenisation of the structure [8,9]. However, some LSM treatments produce no significant effects on corrosion performance [10] and in other cases LSM effects, such as the inhomogeneities produced by overlapping tracks, can degrade corrosion resistance [11].

The aim of the present work was to provide a more detailed understanding of how specific microstructural features result in the observed performance gap between bulk and sprayed Inconel 625. In this study, a number of microstructurally modified versions of Inconel 625 were produced namely: wrought (no oxides, no porosity), thermally sprayed coatings (oxides and porosity), sintered (porosity, no oxides), laser melted wrought (rapidly solidified structure largely free from oxides, no porosity) and laser melted thermally sprayed coatings (rapidly solidified structure with loss of some oxide forming elements, no oxides, no porosity). Potentiodynamic electrochemical testing was carried out in order to investigate links between specific microstructural features and electrochemical behaviour.

2. Experimental procedures

2.1. Materials

Wrought Inconel 625 (UNS N06625) was obtained in the form of 5 mm thick cold rolled annealed sheet from Special Metals Corporation. Gas-atomised Inconel 625 powder for HVOF spraying with nominal size range $-53+20\,\mu m$ was used (Praxair Ni 328-5/T1265F). Table 1 gives the composition of the material in wrought and powder form showing slight differences (mainly in Fe content). The composition of the wrought material is as determined by spark emission spectroscopy whereas that of the powder is from a certificate of analysis provided by the suppliers.

2.2. High-velocity oxy-fuel spraying

A MetJet-II liquid fuel HVOF system (Metallisation Limited, Dudley, UK) was used to deposit the coatings. In the MetJet system kerosene is used as liquid fuel and is combusted with oxygen to produce a hot gas jet into which the powder particles are radially injected using nitrogen as the carrier gas. The operation of the gun and detailed arrangement for spraying are described in detail elsewhere [3]. The majority of the coatings were sprayed onto mild steel coupons ($60\times25\times5$ mm), although a small number were sprayed onto 5 mm thick wrought Inconel 625 sheet for a specific set of corrosion experiments. All substrates were grit blasted and degreased prior to deposition. The spraying parameters used are given in Table 2, typically 30 passes of the spray gun were required at a traverse speed of $1\ m\ s^{-1}$ to achieve coatings with a thickness of approximately 350 μm.

2.3. Sintered samples

The same $-53+20\,\mu m$ Inconel 625 powder used for HVOF spraying was also used to produce the sintered samples. The powder was compacted to produce green pellets by using a single action uniaxial hydraulic press at a compaction pressure of 800 MPa. Prior to compaction, the wall of the 22 mm diameter die was lubricated with lithium stearate mixed with acetone in order to reduce the frictional losses at the wall of the die and also to ensure easy removal of the compacted pellet. 13 grams of Inconel 625 powder were used to form each sample.

The compacted specimens were sintered in a laboratory type silicon carbide resistance heated tubular furnace in a flowing argon environment with a flow rate of approximately $0.2\ l\ min^{-1}$. Flowing argon gas was used throughout the heating, isothermal sintering and cooling of the materials to avoid any form of oxidation. Samples were sintered at either of two temperatures, 1310 °C and 1325 °C, for 60 min. The heating rate was $20\ K\ min^{-1}$ followed by isothermal holding at the selected temperature for 60 min and furnace cooling. Pellets weighing 13 g and 5 mm in height were obtained after sintering.

The density of the sintered samples was obtained by measuring the mass and dimensions of the samples. Porosity was calculated by comparing the density of the sintered samples, ρ_{sample}, with the bulk density of Inconel 625, ρ_{bulk}, as shown in Eq. (1).

$$Porosity = \frac{\rho_{bulk}-\rho_{sample}}{\rho_{bulk}} \times 100\% \quad (1)$$

It should be noted that the pores in the sintered samples are not connected, hence the porosity is referred to as non-interconnected porosity.

2.4. Laser surface melting

Laser melting was carried out by an industrial high power diode laser (HPDL) ROFIN-SINAR DL 025, with maximum power of 2.2 kW and a wavelength of 940 ± 10 nm. The laser head was stationary, with the beam vertically incident on the samples which were clamped to a CNC controlled x–y table. Both wrought Inconel 625 and the thermally sprayed coatings were laser treated within an Ar gas environment. A rectangular beam (6×3 mm) was focused on the surface so that the relative movement of the laser and samples produced ~6 mm wide melt-tracks. The laser power was kept constant at 1500 W, the scan speed was varied in the range of 500-2000 mm min^{-1} in order to generate resolidified structures with a range of secondary dendrite arm spacings.

2.5. Electrochemical tests

Electrochemical performance was evaluated by potentiodynamic tests in an acidic, 0.5 M H_2SO_4, electrolyte. A three electrode cell was employed using methods similar to ASTM G5-94 and G61-86 test

Table 2
HVOF spraying parameters.

Oxygen flow rate (l/min)	Kerosene flow rate (ml/min)	Nitrogen flow rate (l/min)	Powder feed rate (g/min)	Chamber pressure (bar)	Nozzle length (mm)	Spray distance (mm)
910	480	5	80	7.8	100	350