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Thermally Sprayed Inconel 625 Coating on 304 Stainless Steel: A Neutron
Diffraction Stress Analysis

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Abstract

The aim of this project is to enhance fundamental understanding and gaining knowledge about the mechanism and factors controlling the residual stress formation in the Inconel 625 coating on 304 Stainless steel substrates, with potential application to coupled bi-metal(?) materials. It is noted that designing high-performance coatings with long-term active corrosion protection for such components will add value to many strategic industries, including the automotive and aerospace sectors. In this paper, the investigation was conducted to characterise stress and mechanical properties of Inconel 625 with different thicknesses, 300µm and 400µm, deposited on 304 stainless steel (SS) substrate using high-velocity oxy-fuel (HVOF) spraying technique. The coating characterization was carried out to determine the optimal coating thickness to be applied on 304 SS substrates that achieves the best coating performance. The residual stresses were systematically characterized to understand the influence of coating thickness within the coating vicinity using the neutron diffraction technique. The results indicate that the residual stressed were compressive in nature but of slightly different magnitude despite using the same powder as feedstock. This was attributed to the influence of thermal and cooling effect that plays a major role in the coating build-up or some possible random factors.

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1. Introduction

Inconel 625 is a nickel-chromium-molybdenum based alloy with superior corrosion, wear and high temperature resistance [1]. Although, initially developed for high temperature strength, the alloy has found an array of applications in extremely corrosive environments in the aerospace and marine industries. Resistance to different corrosive environments is attributed to certain alloying elements that constitute the alloy matrix; Nickel majorly offers resistance to reducing conditions and neutral and alkaline salt solutions [2] while passivation, as a result of the chromium content within the alloy's matrix shields it from oxidizing conditions [3]. The high mechanical strength of the alloy is credited to the joint action of Molybdenum and Niobium, which stiffens the matrix of the alloy. Moreover, molybdenum offers resistance to pitting corrosion especially to aggressive agents like chlorides and sulphates [1].

Due to economic considerations, most materials are used as a coating rather than a bulk material for the manufacture of component parts used in such industries. Coatings, therefore need to be developed to exhibit superior properties and durability for their intended application. One way of achieving this, is by developing current spraying techniques to create homogenized surfaces of coating structures. Thermal spray coating techniques such as the high-velocity oxy-fuel (HVOF) have been studied, extensively employed and proven to offer excellent resistance to corrosion and wear relative to electroplated coatings [2]. The HVOF technique is usually used when a thick coating, with low porosity and high adhesion is desired. This is because the technique achieves as little degradation as possible with less oxide inclusions and phase transformations [2]. However, in order to achieve superior coating properties, the process should still be optimized, especially the particle velocity during deposition and the flame temperature, which are the major two factors to consider in developing the coat morphology and selecting the spray feedstock, respectively [3].

Controlling the build-up of residual stresses during thermal spray deposition remains crucial in the performance of the coat. Adhesion strength of the coating depends on the nature and magnitude of the residual stresses [4]. Some particles may remain unmelted within the coating material as a result of low deposition temperature resulting in the generation of peening stresses which may cause localized compressive residual stresses on the bond interface. Other stresses that should be considered may arise from thermal differences at the bond interface [4]. All these stresses are most likely to curb crack initiation and propagation if they are of a compressive nature, thereby strengthening the adhesion bond. The quality of adhesion can also be increased by removing any grease or loose particles on the surface of the substrate [2]. This is due to the simple fact that, powder particles easily anchor on rough surfaces, therefore the substrate surface should preferably be of a high degree of roughness.

A lot of factors need to be optimized to achieve high performance coatings. This work is therefore focused on understand the mechanisms and factors influencing the generation of residual stresses; which greatly influence the corrosion performance of coatings.

2. Experimental Procedure

2.1. Samples and spraying conditions

The samples used for this investigation comprised 304 stainless steel substrate and Inconel 65 powder as feedstock for the High-Velocity Oxy-Fuel techniques (HVOF) techniques. Prior to the coating, the 304 stainless steel were grit-blasted with alumina to clean and increase the adhesion of the coating. Coatings of about 300 μ m and 400 μ m in thickness were deposited by TAFA JP500 HVOF on the 304 stainless steel of size 100 x 100 x3mm. The parameters were the same on all coated samples and were as follow: 375 mm stand-off distance; 0.03 m³/h fuel (kerosene) flow rate; 52.39m³/h oxygen flow rate; 7.8 bar chamber pressure. The nozzle length and powder feed rate of 101.6mm and 80 g/mm respectively are used for the spraying.

2.2. Coating characterization

Coating microstructure and phase analysis were studied using scanning electron (SEM) equipped with energy dispersed X-ray spectroscopy (EDS) and X-ray diffraction (XRD) using Cu radiation. The grain sizes were determined with the aids of Scherrer's method.

2.3. Residual stress measurement

Residual stresses within the combine Inconel 625 coatings were determined by the neutron diffraction technique at ANSTO OPAL reactor neutron facility (Australia) using KOWARI residual stress diffractometer [7]. With neutron diffraction technique the method allows full residual stress profiling through the whole thickness of the coating/substrate system with high spatial resolution [8]. A variation of the same method can also allow to reconstruct stress in the coating even when strain/stress measurement in the coatings is not possible (or not efficient, or not accurate enough). This so-called indirect approach was successfully tested and used for stress analysis of WC-Co coating of similar thickness [9]. Discussed shortly in [9], this method utilizing the stress balance principle applied to the substrate/coating stress distribution. This indirect approach was an essential part of the stress analysis because the neutron diffraction peak position of the Inconel 625 and 304 SS were close (approximately 0.3-degree separation). Therefore, when measurements are done in the locations too close to the interface so that two materials contribute to the diffraction signal, the peak separation by peak fitting procedure was impossible. (Generally it is possible with overlapped peaks but when they are separated by at least 1 degree.) As a result, all measurement points affected by this diffraction peak mixing effect in the range of ± 0.3 mm from the interface were so inaccurate or unreliable, that they were discarded. Only in the thinner coating of 0.4 mm thickness few points were not affected.

With focus on indirect approach a series of accurate measurements through the 304 SS substrate were done using wavelength of 1.55 Å obtained from the Si(400) monochromator at the take-off angle of 69°. The γ -Fe(311) reflection at the Bragg angle of 90.6° was utilised during strain measurements with the gauge volume of 0.2x0.2x20 mm³. The strain measurements in the 304 SS substrates were done in steps of 0.2 mm in the two principal directions, in-plane and normal to the surface. The balanced biaxial plane stress assumption was used to recalculate the in-plane stresses from the measured d-spacings, while the normal stress component is assumed to be zero.

The full techniques used have been discussed elsewhere by the current authors [9].

3. Results and Discussion

Figure 1(a) shows the typical morphology of the Inconel 625 powders. The powder was dense with good sphericity. Poza et al [10] reported that powder of such characters was good for thermal spraying, as help in powder flowability.

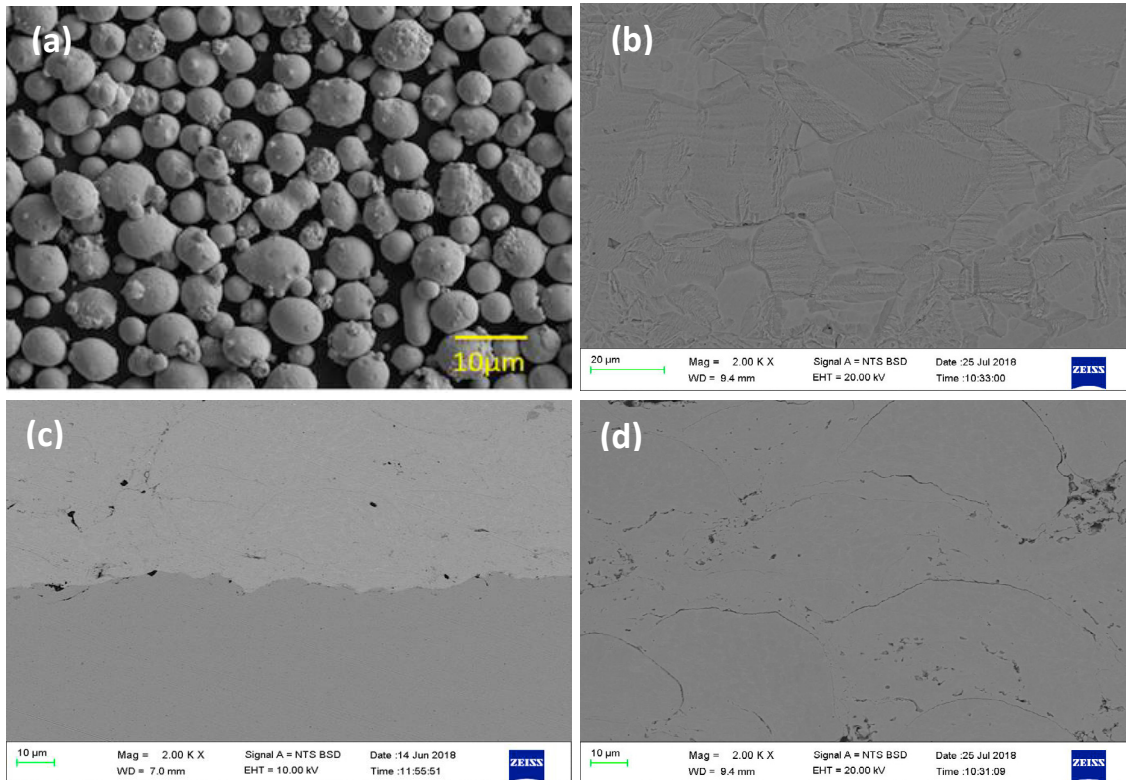


Figure 1. SEM/BSE image of the (a) Inconel 625 powder, (b) 304 SS substrate. The bottom images (c) and (d) show the cross-sectional microstructures of the Inconel 625 coating of thickness 300 μm.

The investigation was further extended to study the integrity of the 304 stainless steel substrate prior to deposition (Figure 1(b)). The image (Figure 1(b)) revealed twin grain boundaries, with less porosity, which is a typical characteristic of a 304 SS substrate. The SEM/BSE of the Inconel 625 coating on 304 SS is shown in Figure 1(c&d). The image shows a typical lamella structure of Inconel 625 coating (At high magnification). The coating reveals low overall porosity with small defects of spherical morphology in the inner parts of splats and the network of inter-splat linear defects formed on the splat boundaries during deposition. Some microcracking were observed on the coating resulted from the influence of residual stresses within the coating vicinity (Fig1 (d)). In addition, Fig1(c) confirm that the coating fitted the substrate surface perfectly, hence would create good mechanical bonding. EDX analysis shows that the Inconel 625 coatings were composed of Ni, Cr, C, Mo (little) and Fe. The presence of Fe at the coating interface is thought to be originated from the substrate. Thus, suggested that diffusion does occur within the coating vicinity. The XRD spectra of the coating are similar, but intensity varies. The major phase identified on the as-sprayed coated sample is Cr-Ni of FCC structure.

The physical properties of the Inconel 625 coating are shown in Table 1. The results show that the coating has high mechanical strength [10], however, their properties are different despite using the same feedstock. This was due to differences in their coating thickness which of no doubt will play a role when using for an engineering application.

Table 1: Inconel 625 coating properties for thickness 300 μm and 400 μm

Material (μm)	Yield Strength (MPa)	Ultimate tensile strength (MPa)	Average Residual stress (MPa)	Grain Size (nm)	The surface roughness (μm)		
					Ra	Rz	Rmax
300 μm coating thickness	1575	1837.11	-307 ± 25	142.2 ± 0.1	18.92	94.10	102.53
400 μm coating thickness	1658	1934.68	-355 ± 25	128.1 ± 0.1	15.98	82.74	96.97

The depth-resolved stress profile measured in the as-sprayed Inconel 625 of thickness 300 μm and 400 μm by indirect approaches are shown in Figure 2, with the summary of the average stress determined in the Inconel 625 coating presented in Table 1

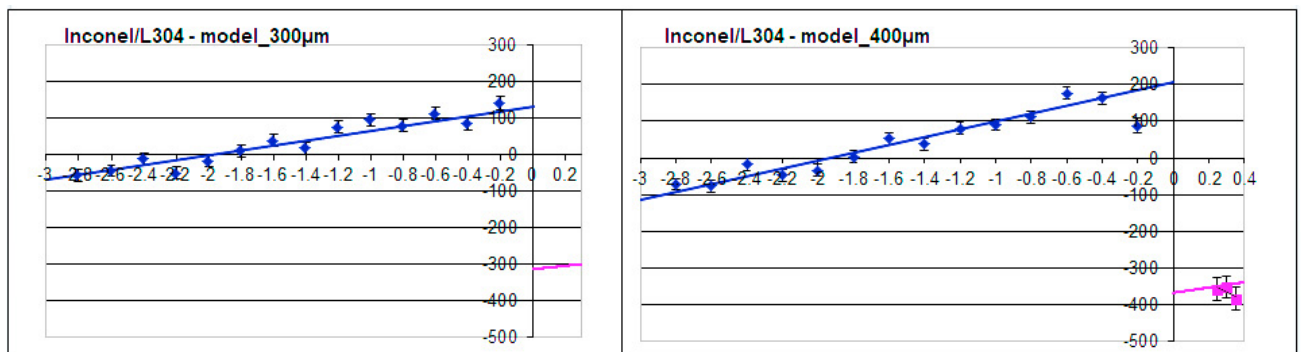


Figure 2. Depth-resolved through the thickness in-plane residual stress profile measured by neutron diffraction in Inconel 625 coating of thickness 300 μm and 400 μm deposited on the 304 SS. The average stress determined in the coatings by employing the direct and indirect approaches outlined in the text is indicated by the data points in the positive x-axis, while the stress in the substrate is on the left (negative x-axis).

The residual stress measured on both coated samples of thickness 300 μm and 400 μm is compressive (moderate) in nature. The formation of compressive stress was attributed to the following; (a) greater influence of peening mechanism over thermal quenching, as a result of high particle velocity of JP5000 spraying [9, 11], (b) differences in the coefficient of thermal expansion between substrate and the coating. The exact contribution of these two sources is determined by spraying parameters and frequently difficult to separate without accurate knowing temperature, velocity, etc. conditions. Although very close in general trend, the two stress profile and average values are slightly different as well as other experimentally measured properties (see Table 1 and Figure 2). Thus, it is suggested that additional factors responsible for this, deterministic or stochastic in nature. It might be thought that one of the major factors for the differences was coating thickness, but, considering theoretically, keeping all other spraying parameters and conditions the same, the increased thickness is, in fact, supposed to decrease the stress value in the coating, therefore it is not just the thickness factor. As a deterministic factor, it might be a small difference in the thermo-mechanical history (e.g. longer deposition time and therefore a different temperature regime leading to different stress states). In addition, it might be some inconsistency in the spraying regime which played a role in the cooling and solidification of the coating that could also result in the deposits with slightly different mechanical properties (UTS and YS), different microstructure (average grain size and roughness) as well as different stresses (Table 1).

Overall, the residual stresses (compressive) increase with an increased in the coating thickness, as well as their yield and ultimate strength, although the discrepancies were small to make a conclusive argument.

4. Conclusion

The coating microstructures are similar despite differences in the coating thickness. The major feature of the residual stresses in the both coatings is its compressive nature, although of slightly different values in the range of -300 to -350 MPa. As an experimental observation, the residual stress increased slightly with an increased in the coating thickness, though interpretation of this second-order effect requires additional investigation.

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