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# Erosion-Corrosion of $\text{Cr}_3\text{C}_2$ -NiCr High Velocity Thermal Spray Coatings

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by

Steven James Matthews

A thesis submitted in partial fulfilment of the  
requirements for the degree of Doctor of Philosophy in Engineering,  
The University of Auckland, 2004.

# The University of Auckland

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**Abstract**

This thesis investigates the mechanisms of erosion-corrosion of  $\text{Cr}_3\text{C}_2\text{-NiCr}$  thermal spray coatings under high temperature, high erodent velocity, turbine conditions. Erosion-corrosion is a generalised wear phenomenon where the combined effect of each degradation mechanism generates more extensive mass loss than the sum of each mechanism acting independently. Previous research has highlighted several theoretical mechanisms under this generalized process, ranging from the erosion induced breakdown of oxide scales in corrosive environments, through to the development of oxide layers in highly erosive environments. Prior to this current work experimental simulation of these mechanisms has focused on bulk alloy materials with well characterised oxidation responses, under conditions of low temperature, low erodent impact velocity and high erodent flux, conditions which are readily generated within laboratory scale rigs and which tend towards the low impact energy conditions encountered within fluidised bed combustors. Few works have addressed erosion-corrosion under simulated turbine conditions of high temperature, high erodent impact velocity and low erodent flux. While comparative trials have been run under such conditions on a purely mass loss basis, little has been presented regarding the microstructural analysis of such degradation, particularly for materials that rely on the industrially relevant, slow growing oxide scales  $\text{Cr}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ .

Thermally sprayed  $\text{Cr}_3\text{C}_2\text{-NiCr}$  coatings are routinely applied to combat wear at high temperature due to the high wear resistance imparted by the hard carbide particles and the high temperature oxidation resistant nature of the  $\text{Cr}_2\text{O}_3$  oxide formed over both phases. However, most published work characterising the erosion-corrosion response of these coatings has been conducted on a comparative basis by contrasting coatings of various composition ratios, deposited by various techniques, with the response of well characterised bulk materials. Little has been presented on the microstructural mechanism of erosion-corrosion of  $\text{Cr}_3\text{C}_2\text{-NiCr}$  coatings, a point highlighted by the limited understanding of the oxidation mechanism of the  $\text{Cr}_3\text{C}_2$  phase, the oxidation mechanisms of the combined composite  $\text{Cr}_3\text{C}_2\text{-NiCr}$  and the influence of the coating splat structure on the oxidation response. While the erosion response of thermal spray coatings and bulk cermets is more widely understood, most works have been conducted under milder conditions than used in the current work. In addition previous works have been conducted primarily on as-sprayed coatings with few works taking into account the effect of heat treatment induced changes in the coating composition and microstructure that occur with extended in-service exposure at elevated temperature.

In addressing the short comings in the current state of knowledge, the aim of this work was to characterise the mechanism of erosion-corrosion of high velocity sprayed  $\text{Cr}_3\text{C}_2\text{-NiCr}$  thermal spray coatings under turbine conditions, incorporating the effect of variation in the composition and carbide distribution with in-flight degradation, variations in starting powder morphology, heat treatment, erosion conditions and exposure temperature.

75 Cr<sub>3</sub>C<sub>2</sub>-25(Ni20Cr) coatings were deposited by Aerospray™ HVAF, GMA Microjet™ HVOF, Stellite Jet Kote™ HVOF and TAFA JP-5000™ HVOF spraying under optimised conditions using agglomerated/sintered and blended powders. The prealloyed powder based coatings, characterised in terms of microhardness, porosity content and phase degradation, were found to exceed the average values of coating quality presented in the literature. The blended powder based coating of this work was comparable with the coating attributes presented in the literature for plasma and HVOF coatings based on this powder morphology. Based on these results the coatings were considered representative of those sprayed industrially and therefore the responses of the samples in this work to oxidation and erosion were considered indicative of the response of industrially applied coatings of this composition in service.

Heat treatment trials were conducted on the Aerospray™ HVAF and Microjet™ HVOF coatings at 900°C in air and argon for up to 60 days to simulate the compositional and microstructural development of these coatings under elevated temperature conditions in service. In the prealloyed powder based coatings, rapid carbide precipitation occurred within the first two days in both coatings to reach the steady state composition of 75-80vol%. Minimal in-flight carbide dissolution in the HVAF coating led to preferential carbide precipitation on the retained carbide grains. In the Microjet™ HVOF coatings, which suffered extensive in-flight carbide dissolution, carbide precipitation occurred as fine precipitates in the carbide-free zones, forming large sponge-like agglomerates. With extended exposure Ostwald ripening led to coarsening of the individual carbide grain size and widespread agglomeration of the carbide grains into an extensive three dimensional network after 30 days exposure, with minimal development out to 60 days. Compositionally, heat treatment led to a dramatic reduction in the supersaturated matrix phase Cr content, with the steady state Cr composition of the Microjet™ HVOF coating exceeding that of the Aerospray™ HVAF coating based on XRD analysis. Cr<sub>3</sub>C<sub>2</sub> was the only carbide detected with heat treatment. Heat treatment of the blended powder based coating led to sintering of the single phase splats. Diffusion of the carbide elements into the matrix phase splats occurred, allowing fingers and nodules of the carbide to develop into this phase as well as increasing the matrix phase Cr concentration.

Oxidation of Cr<sub>3</sub>C<sub>2</sub> by hot stage XRD analysis at 600°C, combined with TGA analysis at 600-850°C, supported the mechanism of stepwise decarburisation prior to Cr<sub>2</sub>O<sub>3</sub> formation, presented as one possible mechanism in the literature. Oxidation of the Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings over the range 700-850°C was dependent on the starting powder morphology and the extent of dissolved carbide in the matrix phase. Oxidation of the as-sprayed prealloyed powder based coatings was dictated by the matrix phase, the high Cr concentration resulting from carbide dissolution leading to rapid growth of the Cr<sub>2</sub>O<sub>3</sub> phase over the oxidising carbide grains. Growth stresses induced by such overgrowth lead to the formation of interfacial voids over the carbide grains at high temperature. Heat treatment reduced the matrix phase Cr concentration, resulting in the coating phases oxidising independently with a reduced magnitude of lateral matrix based scale growth over the carbide phase. In the blended powder based coating, bulbous Ni oxides dominated the scale topography. With extended exposure a continuous Cr<sub>2</sub>O<sub>3</sub> scale formed below the faster growing Ni oxides, which enabled lateral growth of the scale over the carbide based Cr<sub>2</sub>O<sub>3</sub> scales. Following heat treatment the matrix phase Cr concentration increased, minimising the development of Ni oxides on this phase.

Erosion studies were carried out in a custom built high temperature erosion apparatus. Ambient temperature trials were conducted using  $\text{Al}_2\text{O}_3$  erodent at velocities of 150m/s. The as-sprayed prealloyed powder based coatings exhibited a brittle impact response, which was accentuated in the Microjet™ HVOF coating by the increased extent of in-flight carbide dissolution and the splat structure which made this sample more susceptible to brittle erosion mechanisms. Heat treatment of these coatings led to sintering of the splats and a more ductile impact response due to the increased ductility of the matrix phase. The as-sprayed blended powder based coatings exhibited a range of impact responses from brittle erosion of the carbide through to ductile erosion of the matrix based splats. Mass loss was accentuated by the poor intersplat adhesive strength. Heat treatment led to sintering of the splats, resulting in a more microstructural based erosion response.

The two prealloyed powder based coatings generated similar erosion rates under the aggressive conditions, distinctly more erosion resistant than the blended powder based coatings. Heat treatment improved the erosion resistance of all the coatings, however, the duration of heat treatment had a negligible effect on the magnitude of erosive mass loss.

Erosion at 800°C, with an impact velocity of 235m/s, lead to significantly deeper erodent penetration into the coating than noted at ambient temperature. The significant increase in the matrix phase ductility at elevated temperature minimized the impact of carbide dissolution on the matrix impact response in the prealloyed powder based coatings. The primary effect of carbide dissolution was to reduce the carbide concentration, allowing deformation of the matrix to dictate the coating response. Carbide development with heat treatment significantly reduced the ability of the matrix to deform in this manner. The increased matrix phase ductility in the blended powder based coating reduced the concentration of impact energy on the splat boundaries, leading to a more microstructural based erosion response. Heat treatment had a negligible effect on the coating response, given the reduced significance of the splat boundary adhesion.

Erosion at 700°C generated similar erosion responses in the prealloyed powder based coatings to those noted at 800°C, the lower matrix phase ductility reflected in the more brittle response evident as brittle cracking and fracture. The effect of carbide development with heat treatment was not as dramatic as at 800°C due to the reduced matrix phase ductility at this temperature. Erosion of the blended powder based coating at 700°C generated the same spectrum of erosion response as noted at 800°C in both the as-sprayed and heat treated states, with the variation in matrix phase ductility with temperature overshadowed by the heterogeneous coating impact response resulting from the heterogeneous phase distribution.

The steady state erosion rate at 700°C was comparable across all of the coatings in both the as-sprayed and heat treated conditions. At 800°C, heat treatment had a negative impact on the prealloyed powder based coatings, but no definitive effect on the blended powder based coatings. The Microjet™ HVOF coatings were more erosion resistant than the Aerospray™ HVAF coatings under these conditions. These results pointed to a reduction in the significance of the coating splat structure on the magnitude of erosion, in favour of a more microstructural based response at high temperature.

In both the ambient and elevated temperature trials the coating microhardness values proved to be a poor indicator in predicting the magnitude or relative ranking of the erosion response of the different coatings under these aggressive erosion conditions.

Erosion-corrosion under turbine conditions of high temperature, high erodent velocity and low erodent flux, was simulated by oxidizing samples at 900°C and subjecting them to one second of erosion every 48 hours over a period of 60 days. The degradation testing was assessed in accelerated testing in additional trials by polishing the oxide scale formed at 900°C from the sample surface every 48 hours over the same time period. Under these conditions the coatings formed thick oxide scales that penetrated into the coating. Preferential internal oxidation of the Cr<sub>3</sub>C<sub>2</sub> phase occurred in the coating, consuming the grains in the near surface zone through the formation of Cr<sub>2</sub>O<sub>3</sub> to a depth dependent upon the test temperature. Oxygen ingress occurred along the carbide-matrix interface and was accentuated in the regions of impact damage surrounding the erodent indentations. Internal oxidation of the carbide phase sealed off the pockets of matrix phase which were eventually consumed by oxidation once they were no longer able to maintain a protective Cr<sub>2</sub>O<sub>3</sub> oxide. The extent of internal attack was consistent in the 20, 40 and 60 day samples, suggesting that the internal oxidation front proceeded at a constant rate in front of the erosion front into the coating. The prealloyed powder based coatings were more resistant to such internal degradation relative to the blended powder based coatings, with an internally oxidised zone of 6µm relative to the 10µm thick internally oxidized band in the blended powder based coating. While each erodent impact event may be classified as oxidation affected erosion, the low erodent flux effectively led to a long-term response more accurately described as erosion affected oxidation.

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