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# Application of small punch creep testing to a thermally sprayed CoNiCrAlY bond coat



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#### ABSTRACT

High velocity oxy-fuel thermal spraying was used to prepare free-standing CoNiCrAlY (Co–31.7% Ni–20.8% Cr–8.1% Al–0.5% Y (wt%)) bond coat alloy samples approximately 0.5 mm thick. Creep tests were conducted at 750 °C on these samples using a small punch (SP) creep test method. The samples were characterised before and after creep testing using scanning electron microscopy with electron backscatter diffraction (EBSD). EBSD revealed a two phase fcc  $\gamma$ -Ni and bcc B2  $\beta$ -NiAl microstructure with grain sizes  $\sim 1-2~\mu m$  for both phases, which did not change significantly following testing. The constant temperature SP test data were characterised by a minimum creep strain rate,  $\dot{\epsilon}_{min}$ , and a total time to failure,  $t_f$ , at different applied stresses. The data are fitted to conventional power law equations with a stress exponent for creep close to 8 in the Norton power law and between 7 and 10 in the Monkman–Grant creep rupture law. Creep rupture was predominantly due to creep cavitation voids nucleating at both the  $\gamma$ - $\beta$  interphase boundaries and the  $\gamma$ - $\gamma$  grain boundaries leading to final failure by void linkage. However, rupture life was influenced by the quantity of oxide entrained in the coating during the spray deposition process.

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#### 1. Introduction

The continuing requirement to improve the efficiency of aeroengine and land based gas turbines means that there is a need to continue to increase turbine gas inlet temperatures. Thus thermal barrier coating (TBC) systems, which are used to protect turbine blades from these harsh operating environments, continue to be intensively studied so that their durability can be improved [1-3]. Generally a TBC system is deposited on a superalloy substrate and consists of an aluminium containing bond coat (BC) between the superalloy and the ceramic top coat, which acts as the actual thermal barrier. Additionally, a thermally grown oxide (TGO), predominantly alumina, forms at the interface between the bond coat and the topcoat during service at elevated temperature. The chemical, microstructural and mechanical characteristics of the bond coat are crucial to the durability of the overall system. This is because oxygen permeates through the ceramic top coat and oxidises the bond coat, slowly thickening the TGO during service [4–7]. This causes a progressive build-up of stress in the system and eventual spallation of the ceramic coating. It is the way the strain from TGO growth is accommodated by the creep, yield and ductility characteristics of the bond coat that will often determine the coating lifetime [8,9]. Bond coats are typically either of the diffusion aluminide type or the overlay MCrAlY-type (M=Co, Ni). Overlay coatings, deposited by low pressure plasma spraying (LPPS), vacuum plasma spraying (VPS) or, more recently by high velocity oxy-fuel (HVOF) thermal spraying, have become more widely used because of advantages such as lower cost, better control of composition and the ability to employ complex alloys very different in composition from the superalloy substrate. MCrAlY alloys are typically complex multi-phase materials and can comprise, for example, fcc  $\gamma$ -Ni and bcc B2  $\beta$ -NiAl phases or  $\gamma$ ,  $\beta$ and ordered  $\gamma'$  (Ni<sub>3</sub>(Al,Ti)) phases and can have a composition which is tailored to achieve specific performance needs in particular applications [10]. Furthermore, the thermally sprayed bond coat microstructure depends critically on the following factors: the alloy composition; the type of thermal spray process used; the process parameters employed and the overall coating thickness deposited. All of these factors can affect the mechanical properties of the coating. It is therefore important to distinguish between the mechanical behaviour of bulk MCrAlY alloys and that of MCrAlY coatings produced by industrially relevant thermal spray processes and with thicknesses relevant to TBC applications (typically  $\sim$  200  $\mu m$  thick). Published work on the high temperature mechanical properties of MCrAlY alloys is somewhat sparse, despite the importance of their creep and yield properties to the durability of TBC systems and failure by spallation. Early work by Smith [11] on a thick free-standing CoCrAlY alloy prepared by LPPS

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demonstrated that it showed ductile-to-brittle transition behaviour around 700 °C but over 70% elongation in a tensile test at 850 °C. Later, Hebsur and Miner [12,13] undertook constant load creep rupture tests on samples of a 9 mm thick NiCoCrAlY (PWA 276) alloy at 660 °C and 850 °C also prepared by LPSS. They too observed a ductile-to-brittle transition (at  $\sim 600$  °C), creep behaviour between 660 and 850 °C, and strain rate sensitive superplasticity above this temperature. Stress relaxation experiments were carried out by Wereszczak et al. [14] using bulk MCrAlY alloys prepared by hot isostatic pressing of gas atomised powders to determine creep parameters whereas Clyne and co-workers [15] used the VPS method to make 1.4 mm thick samples of NiCrAlY and CoNiCrAlY alloys for creep testing of miniature tensile samples at 750 and 850 °C. Primary, secondary and tertiary creep regimes were noted and steady state creep rates derived from their experiments. Also Taylor et al. [16] reported a method for the evaluation of the creep properties of as-deposited MCrAlY coatings by using a composite tensile specimen in which the overall strain/time response had to be deconvoluted to obtain the creep characteristics of the coating. They claimed that the thin coating behaved differently from a bulk alloy of the same chemistry.

Generally, researchers have fitted their creep data to the well-known power-law creep equation:

$$\dot{\varepsilon} = A\sigma^n \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where  $\sigma$  is the stress, n is the stress exponent, Q is the apparent activation energy for creep, R is the gas constant, T is the absolute temperature, A is a constant and  $\dot{e}$  is the steady state creep rate. However, there is wide variation in the values of n, A and Q that are reported. Bose [17] provides a useful summary of the somewhat limited data on both ductile-to-brittle transition and creep of bond coat materials.

Given the importance of bond coat creep in influencing the durability of TBC systems there is clearly considerable need to determine creep properties of samples which are prepared by thermal spraying processes such as HVOF or LPPS and which have thicknesses as close as possible to those used in industrial TBC systems. One method of testing which appears to have the potential to provide such measurements is the small punch (SP) creep test. The SP test technique [18] is based on the deformation of a miniature disc-shaped test specimen typically 8-10 mm in diameter and around 0.5 mm thick. The test is carried out by applying a loaded ball to one surface of the disc which is firmly clamped around its rim. The response to applying a constant load over a period of time is then recorded to obtain creep displacements. The SP test for creep behaviour involves complex deformation processes [19]. Nevertheless, a recent review [20] has indicated that SP creep testing can lead to consistent results providing jig and specimen geometry are carefully controlled. Hitherto, the SP test has been most widely used for assessing creep behaviour of bulk steels used in nuclear and conventional power plant [21] where its ability to characterise in-service materials using only a small volume of material has been of significant benefit. More recently, it has been shown capable of analysing different zones of weldments in both creep resistant steel [22] and a nickel-based alloy [23]. However, SP testing of MCrAlY bond coats does not appear to have been previously

Overall, the advantage of using the SP test is that data can be obtained from a miniaturised sample obtained directly by thermal spraying (the manufacturing process used in TBC production) and with a thickness approaching that of bond coats used on gas turbine components. This is important given the sensitivity of the bond coat microstructure to even small changes in the many thermal spray process parameters [24]. Minor changes in

microstructural features, such as volume fraction and size distribution of oxide inclusions, will in turn be expected to significantly affect the creep properties. This advantage has to be set against the fact that the SP test involves complex loading and deformation behaviour as first described by Chakrabarty [25] and more recently analysed using finite element models [19]. Although, the SP test does not currently have a universally accepted route for converting the results to equivalent uniaxial test data on full sized samples there is on-going work using finite element modelling to address this [26]. Moreover, it can be argued that this limitation does not detract from the value of the test for bond coats as these are not manufactured from conventional bulk material. It is more important that the SP test is able to generate reproducible and reliable comparative data on the creep behaviour of coatings prepared with different chemical compositions or by different thermal spray processes (e.g. low pressure plasma spraying versus high velocity oxy-fuel thermal spraying).

The aims of the work reported in this paper are to determine if the SP test is a suitable method for examining creep behaviour of thermally sprayed bond coats, to investigate the microstructural changes and failure characteristics in the test and to quantify the creep behaviour in terms of creep rupture times and well known power law relationships. SP tests were carried out at 750 °C on samples of free-standing HVOF thermally sprayed coatings, approximately 0.5 mm thick, produced from a commercially available CoNiCrAlY powder. Typically, bond coats operate in the temperature range 700-950  $^{\circ}\text{C}$  [1] and because the present study was aimed, in part, at determining the suitability of the SP test for MCrAlY alloys the temperature chosen was towards the lower end of this range. This is well within the capability of the existing rig (designed principally for work on high temperature steels) whilst still being applicable to bond coats in service. It is notable that the sample thickness used in this work was significantly closer to that of alloy bond coats used in industrial TBC systems than has normally been the case for previously reported studies on MCrAlY alloys.

#### 2. Experimental

#### 2.1. Materials, HVOF thermal spraying and heat treatment

The coatings used in the creep experiments were prepared by HVOF thermal spraying using powder with the following nominal composition Co–31.7% Ni–20.8% Cr–8.1% Al–0.5% Y (wt%). The powder was obtained from Praxair (CO-210-24) and had a size range of  $-45+20\,\mu m$  with a chemically analysed oxygen content of 0.037 wt% O. The coatings were deposited onto mild steel substrates with dimensions  $60\times25\times1.8~mm^3$  using a Met Jet III liquid fuel HVOF gun. The details of the spraying process and procedures are given elsewhere [27]. Two sets of coatings were produced for this study in two separate spray runs and hereafter are referred to as C1 and C2. The primary spray parameters employed were nominally identical for the two runs. Coatings were sprayed to a thickness of approximately 0.5 mm and were then debonded from the mild steel by bending around a mandrel.

Detached coating samples were vacuum heat treated at 1100 °C for 2 h followed by natural cooling, to replicate the initial heat treatment given to bond coat alloys during TBC manufacture. Previous work has shown that such a heat treatment is sufficient to reduce to a minimal level any porosity that might be present in these coatings after HVOF spray deposition [27]. Specimens 8 mm diameter in the form of discs for SP testing were cut from the heat treated coatings by electro-discharge machining. They were ground down from the as-deposited thickness and polished to a final thickness of approximately 430  $\mu$ m using 1  $\mu$ m diamond

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