# 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 ~  $1-2 \mu m$  for both phases, which did not change significantly following testing. The constant temperature SP test data were characterised by a minimum creep strain rate,  $\dot{\varepsilon}_{min}$ , and a total time to failure, t<sub>f</sub>, 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.

*Keywords*: HVOF thermal spraying; MCrAlY bond coat; mechanical testing; creep; phase transformation

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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 Additionally, a thermally grown oxide (TGO), predominantly thermal barrier. 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 y-Ni and bcc B2 β-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  $\sim 200 \,\mu\text{m}$  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 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 ~ 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 MCrAIY 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 NiCrAIY and CoNiCrAIY 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 MCrAIY 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  $\dot{\epsilon}$  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 MCrAIY bond coats does not appear to have been previously reported.

Therefore 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 on samples of free-standing HVOF thermally sprayed coatings, approximately 0.5 mm thick, produced from a commercially available CoNiCrAlY powder. The sample thickness used here was thus significantly closer to that of alloy bond coats used in industrial TBC systems than has 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  $60 \times 25 \times 1.8$  mm using a Met Jet III liquid fuel HVOF gun. The details of the spraying process and procedures are given elsewhere [24]. 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 [24]. 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 paste. The final thickness was controlled to within ± 8  $\mu$ m as measured by a digital micrometer.

#### 2.2 Small punch creep test

The small punch tests were carried out in line with the guidance provided in the CEN Workshop Agreement, CWA 15627:2006 E [25], using the equipment shown schematically in Fig. 1. It comprises a steel frame, A, a sample holder, B, which supports the sample and a clamping head, C. A hemispherical, high temperature steel, punch, D, is positioned co-axially with respect to the axis of the sample. The load is placed on the pan, E, and is transmitted to the sample via the vertical rod, F. The sample is maintained at a constant temperature by the furnace, G, and displacements are measured through the punch movements and recorded by a LVDT transducer, H, to an accuracy of  $\pm 1 \ \mu m$ .

In the present work, SP creep tests were carried out by employing loads ranging from 67.5 to 100 N at 750 °C in air. For each creep test, the displacement of the centre of the disc,  $\delta$ , was recorded as a function of time.

Load can be converted to stress using equation (2), derived from membrane stretching theory [25], where  $a_p$  is the distance between the punch axis and clamping edge,  $a_p=2$  mm in all experiments reported in this paper.  $R_s$  is the radius of the punch, which is 1.25 mm and  $t_0$  is the thickness of the disc, which is 0.430 mm.  $K_s$  is a nondimensional correction factor to take account of localized necking during large strain deformation. It is believed to be material dependent and for steels is typically around 1.2. In the present work load was converted to stress using equation (2) and taking  $K_s$ to be 1.

Equation (3) [26] relates the strain,  $\varepsilon$ , to the displacement of the punch,  $\delta$ . The displacement rate can be converted to strain rate using equation (4), obtained by differentiating (3).

$$\sigma = \frac{P}{3.33 \times K_s \times a_p^{-0.2} \times R_s^{1.2} \times t_o}$$
(2)

$$\varepsilon = 0.15 \left(\frac{\delta}{t_0}\right)^{1.5} \tag{3}$$

$$\dot{\varepsilon} = 0.225 \left(\frac{\delta}{t_0}\right)^{1.5} \left(\frac{\dot{\delta}}{\delta}\right) \tag{4}$$

#### 2.3 Material characterisation

The microstructures of coating cross-sections before and after testing were examined. Small samples were cut, mounted in conducting resin, ground and diamond polished to a 1  $\mu$ m finish. In the case of samples taken following testing, these were cut from different regions so that deformed and undeformed sections could be studied i.e. reference samples were taken from the clamped region where the alloy had been at elevated temperature but had not undergone creep.

Prepared samples were examined in a FEI XL 30 scanning electron microscope (SEM) operated at 20 kV. Both backscattered electrons (BSE) and secondary electrons (SE) were used to form images and semi-quantitative energy dispersive X-ray analysis (EDX) was utilised to aid phase identification through chemical microanalysis. Electron backscattered diffraction (EBSD) was used to investigate grain morphology before and after creep testing. Samples for EBSD required a further stage of chemical/mechanical polishing using colloidal silica to achieve a surface finish of 0.02  $\mu$ m with the minimum surface deformation. SEM-based EBSD was carried out on a Zeiss 1530 VP field emission gun scanning electron backscattere diffraction system (EDAX, Mahwah, NJ, USA). The EBSD patterns were recorded at a specimen tilt angle of 70° with an accelerating voltage of 20 kV and a beam current of 26 nA. EBSD mappings of the specimen were collected with dimensions 35  $\mu$ m × 25  $\mu$ m at a step size of 0.1  $\mu$ m.

The oxygen contents of the sprayed coatings were determined by inert gas fusion combustion analysis of debonded material at an external laboratory.

#### 3. **Results**

#### 3.1 Microstructure of the coatings prior to testing

The microstructures of the vacuum heat treated coatings C1 and C2 prior to testing are shown in Fig. 2. It is evident from Figs. 2(a) and (c) that there is very little porosity apparent in both of the samples. As previously reported [24], this CoNiCrAIY coating has a two phase microstructure, consisting of the dark contrast bcc- $\beta$  (Co,Ni)Al phase and the light contrast fcc- $\gamma$  Ni-rich matrix phase. The volume fraction of the uniformly distributed  $\beta$  phase was found by image analysis to be around 33% in both C1 and C2, with an average  $\beta$ -phase size of 1- 2 µm.

Micron sized, dark contrast particles are also visible in Figs. 2(b) and (d). These tend to occur around powder particle boundaries as either discrete particles or thin stringers. Indeed they delineate particle boundaries and reveal the deformed and partially flattened nature of the sprayed powder particles which were originally spherical. EDX analysis showed them to be rich in oxygen and aluminium and they are presumably aluminium oxide which has formed by oxidation of powders in-flight during the HVOF spraying process. From the figures it is clear that their volume fraction is less than 1% in both C1 and C2. However, in C1 there is a more concentrated band of these features visible in the lower half of the coating as seen in Fig 2 (a). Samples of the debonded coatings were chemically analysed to determine their oxygen contents which were found to be 0.40 and 0.36 wt% O for C1 and C2 respectively and this difference is consistent with the microstructural observations.

Figure 3 shows EBSD - derived inverse pole figure and phase distribution maps for vacuum heat treated coating C1 prior to testing. These figures reveal that the  $\beta$ -phase dimensions are typically 0.8-2 µm as noted previously and that  $\beta$ - regions appear to be monocrystalline, The  $\gamma$ -phase is clearly polycrystalline with equiaxed grains that display a wide range of grain diameters typically from 2 µm 500 nm or less. Many of the  $\gamma$  grains are also twinned. There is no evidence for preferred orientation in either the  $\beta$  or  $\gamma$  phases.

#### *3.2 Creep behaviour*

Typical small punch test creep curves from tests of C1, showing the increase in displacement with time, are seen in Fig. 4. It can be observed that there is a large initial and primary component of deformation, an approximately linear, secondary, region with a gradient,  $\delta_{min}$ , which is the minimum displacement rate. Finally, an accelerating tertiary region leads to fracture. These curves show a similarity to typical uniaxial creep curves which normally exhibit primary, secondary and tertiary creep regions. The constant temperature SP test data can also be characterised by a minimum creep strain rate,  $\dot{\varepsilon}_{min}$ , and the total time to failure,  $t_f$ , at different applied stresses. These values are usually represented graphically on plots of  $\log(\dot{\varepsilon}_{min})$  versus  $\log(\sigma)$  and  $\log(\sigma)$  versus  $\log(t_f)$ , as depicted in Figs. 5(a) and 5(b), respectively; the  $\sigma$  and  $\dot{\varepsilon}_{min}$  values are obtained using equations (2) – (4). Linear relationships have often been obtained in these types of plots for various steels and other alloys using the Norton power law at constant temperature (Eq. (5)),

$$\dot{\varepsilon}_{min} = B\sigma^n \tag{5}$$

and the Monkman-Grant rupture power law (Eq. (6)),

$$t_f = \frac{1}{M\sigma^{\chi}} \tag{6}$$

In the present case, data for C1 and C2 (Fig. 5(a)) are found to lie on a common Norton power law best fit line and the parameters B and n are given in Table 1. In the case of the rupture power law, there is evidently a displacement of the data sets for C1 and C2 (Fig. 5(b)) and each coating has been separately fitted to the rupture power law (equation 6) with the parameters, M and  $\chi$  given in Tables 2; for an equivalent stress level, coating C1 fails at a shorter time.

#### 3.3 Post-test microstructural characterization

Fig. 6 shows typical secondary electron images of the fracture surface of coating C1 tested at 50 MPa. The dome-shaped nature of the failed disc is evident in Fig. 6(a). There is no evidence for any brittle cracking and extensive deformation is evident which is consistent with the punch displacement of ~ 1.5 mm at this stress. At higher magnification, Figs. 6(b) and (c), there is evidence of cavitation, ductile tearing of the  $\gamma$ -matrix and a variety of rounded and angular particles are evident on the fracture surface; the latter are presumably the  $\beta$  phase. To investigate creep failure further a limited number of samples were loaded in the rig but then removed prior to final failure. Fig. 7(a) shows a low magnification cross-section of a typical specimen which underwent creep deformation at a stress of 40 MPa but was not tested to final failure; in this case the test duration was 1000 h. Material thinning and the formation of macroscopic cavities are observed in area B. This is the region in which fracture was observed in samples which were allowed to fail. Creep rupture was thus observed to

be occurring in material located at approximately the edge of contact between the specimen and the spherical punch.

In the clamped region, i.e. to the right of region A, the microstructure was found to be essentially unchanged in terms of the  $\beta$ -phase size and morphology and the  $\gamma$ : $\beta$ phase proportions. This indicates that exposure to 750 °C for the duration of the test did not, in itself, induce significant microstructural changes. The grey contrast features in between regions A and B were found to be cavities with extensive internal oxide brought about by oxidation during testing which was undertaken in air, Fig. 7(b).

Higher magnification micrographs of the region A and B are shown in Figs. 7(c) and (d) respectively. There is some evidence that the fine oxides formed during thermal spraying align as a result of the creep deformation as shown by the arrows. In Fig. 7(d) it is evident that local voids are initiated in region B and appear to have linked together to form rather larger cavities seen in Fig. 7(b). 8

Figure 8 shows both an EBSD – derived inverse pole figure and a phase map of a section taken from region B shown in Fig. 7. It is clear that  $\beta$ -phase regions have not coarsened significantly during the test and that  $\beta$  has remained monocrystalline with no preferred orientation. Similarly the  $\gamma$ -phase grains show little change in that they have remained equiaxed, of similar diameter range and have not developed a preferred orientation. Creep failure seems to be occurring predominantly along both  $\gamma$ - $\gamma$  grain boundaries and  $\gamma$ - $\beta$  interphase boundaries. There is no evidence for preferential cracking of the  $\beta$  phase, intragranular failure of the  $\gamma$ -phase or plastic elongation of either phase in the direction of deformation.

#### 4. Discussion

#### 4.1 Relationship to uniaxial creep testing

Small punch creep tests at various loads on both C1 and C2 confirm that at 750 °C creep deformation occurred and produced displacement curves typical of the SP test [19]. As has been previously stated [19], these curves are only superficially similar in form to those typically seen in uniaxial tests. The tertiary creep region of a uniaxial test typically begins at a creep strain of 1- 5% for most materials. In contrast, the equivalent feature in a SP test, when the minimum deformation rate occurs (at a deformation of 1 mm or more), is seen to be at strains of approximately 30% or more as indicated by equation (3). Hence, the minimum displacement rate in SP tests should not be considered to be directly related to the secondary creep region of a uniaxial creep test. The secondary region in the data from a SP test is a result of a balance between the "stiffening effects" of the reducing cone angle which develops during the test and the increasing contact area between specimen and indenter and "accelerating effects" of the material being in the tertiary creep region as well as the thinning of material in the contact region, as deformation increases. Therefore, the minimum displacement rate,  $\delta_{min}$ , is related to the tertiary creep region of the material being in the tertiary creep region of the minimum displacement rate,  $\delta_{min}$ , is related to the tertiary creep region of the minimum displacement rate, the minimum displacement rate in the contact region, as deformation increases.

material, but is also affected by the large strain, changing cone angle and changing contact conditions which occur in the secondary region of the SP test curve as discussed in detail in reference [19]. As can be seen in Fig. 7, the most extensively deformed regions are approximately half way down the wall of the deforming disc. This is consistent with the deformation shapes and equivalent creep strain distributions produced by Ma et al.'s finite element modelling of the SP test [27]. In spite of these limitations, it is generally agreed that uniaxial creep results correlate well with SP results for the wide range of high temperature alloy steels studied

hitherto and that SP creep test data can be usefully used to estimate material creep constants from the well known creep laws (equations (5) and (6)). The log-log plots in Fig. 5 show that the results obtained from SP testing of these HVOF CoNiCrAlY coatings also fall on straight lines, indicating that their constant temperature creep behaviour at 750 °C can be described by the Norton and Monkman-Grant laws. Hence, even if there are limitations in relating SP data to uniaxial creep test data the SP method is a valuable tool for assessing and, quantitatively comparing, creep behaviour of thin, thermally sprayed bond coat alloys.

#### 4.2 Creep fracture of the bond coat samples in the SP test

The log-log plot of minimum strain rate versus stress (Fig. 5(a)) shows that results from both coatings fall on the same straight line, indicating very similar material behaviour during creep deformation. This is not, in itself, surprising given the essentially identical microstructures of the two coatings. However, some difference in behaviour of the two coatings is seen in the plot of stress versus time to failure (Fig. 5(b)) where coating C1 fails at shorter times for a given stress. Coating C1 was found to have a marginally higher overall oxygen content by chemical analysis, 0.40 compared with 0.36 wt% O and, as can be seen from Fig. 2(a), there is a visible band of material in the middle of C1 which was particularly rich in oxide stringers which would account for the higher oxygen level in chemical analysis. The formation of such a band is possibly due to a temporary perturbation in the HVOF process. It seems possible therefore that this microstructural difference between the two coatings is somehow responsible for the earlier creep failure of C1 as discussed below.

Consider first the lack of any shape change of the monocrystalline  $\beta$  phase particles and the retention of the fine equiaxed grain structure in the  $\gamma$ -matrix. These features indicate that deformation in the SP test of the CoNiCrAIY HVOF coating is dominated by creep of the  $\gamma$ -matrix (Figs. 7 and 8). Extensive tertiary creep as seen here is generally due to a high density of mobile dislocations which increases with strain. However, the alloy eventually fractures because of the gradual evolution of grain boundary cavitation. Indeed Figs. 7(c) and 8(b) provide evidence for creep cavitation voids nucleating at  $\gamma - \beta$  interphase boundaries as well as at  $\gamma - \gamma$  grain boundaries. It is likely that once nucleated the voids continue to grow leading to final failure by void linkage. The extensive internal oxidation of voids seen in Fig. 7(b) supports this view. It is important to recall that at 750 °C the  $\beta$ -NiAl phase has little ductility [28] and brittle second phase particles have previously been noted as preferential sites of void nucleation during creep of, for example, duplex stainless steel [29], Nimonic 80A [30] and SiC reinforced aluminium [31].

Although it is proposed that creep rupture is primarily initiated by voids forming at  $\gamma$ - $\beta$  or  $\gamma$ - $\gamma$  interfaces, the fine scale oxide particle and stringers may also have a role in initiating voids. The fine oxide particles are generated during thermal spraying and tend to form at interparticle boundaries ie where feedstock powder particles come into contact. It may thus be expected that these fine oxides decrease creep rupture time as they are also essentially hard, undeformable particles but on a finer scale than the  $\beta$ -phase. Creep failure would then be expected to be accelerated in a coating with a higher concentration of such particles (particularly if somewhat localised) as is the case in sample C1. The net effect would be a shorter creep life as is indeed observed in C1. This seems intuitively correct since a larger volume fraction of hard phase particles gives rise to a greater particle/matrix interfacial area providing more nucleation sites for creep cavitation voids.

#### 4.3 Comparison with previous studies

The limited data available from the literature appear to show that creep parameters of MCrAlY materials depend very significantly on alloy composition, test temperature and the method used to fabricate the test samples. A ductile-to-brittle transition behaviour is consistently observed; it is composition dependent but typically occurs around 450 to 600 °C. Between about 650 and 1050 °C, creep appears to follow Norton and Monkman-Grant power law behaviour [11-17]. It is clear that in the present alloy the ductile-to-brittle transition is below 750 °C, the fracture surface seen in Fig. 3 shows no evidence for brittle fracture and the fine grained fracture surface is entirely consistent with creep failure. With regard to the values of the stress exponent, n, in the Norton power law equation the available data are limited but values in the range 3-5 are suggested [13-15] compared to the present value 7.9. However, previous studies have generally either been conducted at temperatures above that used here (750 °C) or involved different MCrAlY compositions. Hebsur and Miner [12] suggested that power law (dislocation) creep is the governing mechanism at lower temperature in these type of alloys and the stress exponent is usually predicted to vary from 3 to about 10 for such behaviour so the present results could be consistent with this mechanism. However, the grain size of previous samples was not reported and the very fine  $\gamma$ -phase grain size of the present materials could significantly influence the creep mechanisms in the test and the resultant values of the power law parameters. Although it is clear that further work is needed with the SP test to establish with greater certainty power law parameters for specific alloys, it is evident that the test is valuable as a tool for ranking the behaviour of different MCrAlY alloys in the form of thin, thermally sprayed bond coats.

#### 5. Conclusions

- In this work it is shown that the small punch test is a suitable method for determining the creep behaviour of free-standing HVOF thermally sprayed MCrAIY coatings. When compared with the testing of monolithic bulk samples of the coating alloy, this method has an advantage in that the thin samples exhibit similar microstructural features to those of bond coats in service.
- 2. The Norton power law parameter, n, (equation 5) for two separate coatings (C1 and C2) sprayed in different experiments, but with nominally identical parameters, was determined to be the same within the range of test conditions employed; the value of n is approximately 8.
- 3. Different rupture power law parameters were determined for C1 and C2 over the range of stress studied at 750 °C. The coating C1 with the higher oxide content is found to fail at shorter times for a given stress.
- 4. The creep rupture is primarily 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, oxide particles could also play a role in creep failure by acting as additional void nucleation sites.

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#### **Figure Captions**

#### Figure 1.

A schematic diagram of the small punch test rig showing an overall view and an enlarged view of the sample holder, B.

#### Figure 2

Back scattered electron micrographs showing the microstructures of the vacuum heat treated samples prior to testing. (a) low magnification and (b) high magnification of sample C1; (c) low magnification and (d) high magnification of sample C2.

#### Figure 3.

(a) Inverse pole figure map and (b) phase distribution map derived from ESBD data of heat treated coating C1 prior to creep testing. The  $\beta$ -phase is coloured red and the  $\gamma$ -phase green in (b).

#### Figure 4.

Typical small punch test displacement curve at 750 °C of coating C1 for three different loads

#### Figure 5

Log-log plots of (a) minimum strain rate versus stress and (b) stress versus time to failure for C1 and C2 in the small punch creep test. Experimental data are represented by symbols and best fitting lines obtained using equations (5) and (6)

#### Figure 6

Fracture surface of C1 at a stress of 50 MPa and 750 °C: (a) overall view of sample fracture. (b) and (c) fracture surface and higher magnification showing evidence of cavitation, ductile tearing and variety of rounded and angular particles.

#### Figure 7

(a) Low magnification BSE image of a cross section through a sample removed from the test rig prior to final failure ( 40 MPa for 1000 h) showing cavity formation between A and B and final fracture beginning close to B.

(b) Higher magnification image of region near B showing grey contrast features are oxide-filled creep rupture cavities.

(c) and (d) are BSE images of cross sections taken from areas close to regions A and B respectively. The arrows on (c) show some alignment of the micron sized oxides whilst in (d) small voids are visible (early stage creep rupture).

#### Figure 8.

(a) Inverse pole figure map and (b) phase distribution map derived from ESBD data of a section taken from a creep tested sample corresponding to region B in Fig. 7. The  $\beta$ -phase is coloured red in (b) and there is no evidence of preferred orientation in (a).

Figures



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### Figure 2

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## Table(s)

# Table 1

# Norton power law parameters for C1 and C2 (combined data sets)

	B (s⁻¹ MPa⁻'n)	n
C1	6.0×10 <sup>-20</sup>	7.9
C2		

# Table 2

# Rupture power law parameters for C1 and C2 (individual data sets)

	M (h <sup>₋1</sup> MPa <sup>₋</sup> <sup>x</sup> )	χ
C1	3.3 ×10 <sup>-20</sup>	10.5
C2	1.2 ×10 <sup>-15</sup>	7.6