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Measurement of Ductile Brittle Transition Temperature of Coatings Using the Small Punch Test

S R J Saunders, J P Banks and M Wright
Materials Centre
National Physical Laboratory
Teddington, Middlesex, UK, TW11 0LW

ABSTRACT

The small punch test has been developed to measure the ductile brittle transition temperature (DBTT) of corrosion and oxidation resistant coatings used to protect gas turbine blades. The test is carried out by tensile deformation of small thin samples of the coated material using a punch pressed into the substrate alloy. The sample is held in a small jig which itself can be placed in a furnace to allow the test to be carried out as a function of temperature. Vertical displacement of the sample is directly monitored by use of a linear variable transducer, and the onset of coating cracking determined by monitored acoustic emission signals. Results are presented for a number of different MCrAlY and Pt aluminised coatings. In general, as expected, the MCrAlY coatings had lower DBTTs than the Pt aluminised coatings.

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National Physical Laboratory
Teddington, Middlesex TW11 0LW, UK

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1 INTRODUCTION

Coatings designed for use in the hot section of a gas turbine are required to withstand high temperatures and mechanical stressing. A coating is used for either corrosion or thermal resistance, and will be effective only if it remains intact. Thus the mechanical properties of the coating can have a marked effect on the performance of the system, and data is required to provide designers with assurance that the coating system will be fit for purpose. Additionally, during service ageing effects occur due to oxidation and interdiffusion that will alter the coating properties from the as-produced values. It is important, therefore, to be able to measure the properties of the coating in the aged state and this may involve testing small samples taken from a blade that is to be refurbished.

The fracture strain that a coating can tolerate clearly changes as a function of temperature. Brittle behaviour is frequently observed at low temperatures, and, as the temperature is increased, the material becomes increasingly ductile, and in some materials the transition is relatively well defined so that a ductile brittle transition temperature (DBTT) can be defined. In practice, the information needed by the designer should define conditions under which a coating will crack when subjected to the anticipated service stresses. An approximation used by some turbine manufacturers is that the coating should be able to withstand 1% strain [1]. In this case, therefore, the DBTT corresponds to the temperature below which a strain of 1% would result in cracking the coating. The DBTT also becomes important when considering thermal mechanical fatigue (TMF) resistance. In this case due to temperature gradients along the length of a blade produce a hot spot in the centre is found. The material at this point is constrained from expanding by the cooler material surrounding it, and thus is stressed compressively. At high temperatures creep can relieve this stress because the coating creep resistance is generally lower than that of the substrate. On cooling, depending on the extent of the creep relaxation, and differences in thermal expansion coefficients, the coating can be placed in tension, and will crack if the coating reaches its failure strain (which is temperature dependent).

The small punch test has been used to measure the fracture behaviour of bulk and coated samples [2,3,4], and is ideally suited to extraction of samples from service-exposed components. Indeed, the test was first developed to allow examination of small volumes of irradiated materials, and samples as small as 3 mm diameter have been used [2].

2 MATERIALS

Coatings were deposited on IN738, CMSX4 and Rene 80 substrates. Work was carried out on MCrAlY and Pt aluminised variants supplied by Siemens, Chromalloy UK and partners in the European collaborative project on Ultra Efficient Low Emission Power Plants (COST522). The following systems were investigated:

CMSX4/RT22LT
CMSX4/Pt aluminised (2 types)
IN738/Sicoat 2231 (extracted from a turbine blade)

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CMSX4/Sicoat 2453
IN783/Sicoat2453
Rene80/PVD MCrAlY (3 types)

Rolls Royce, Alstom Power and COST 522 partners provided the substrate alloys. All coating systems were tested after receiving the appropriate heat treatment.

All samples used in these tests were 10 x 10 x 0.5 mm and were spark machined to shape from either thicker disc samples or in one case taken from a gas turbine blade that was supplied by PowerGen.

3 EXPERIMENTAL

A jig was constructed from the Ni-based superalloy, IN738, as schematically illustrated in Figure 1 in which the samples were fitted within a recess. A 2.4 mm diameter Si_3N_4 ball mounted into the loading train of a bench-top Instron electro-mechanical test machine was pressed into the centre of the sample that was located above a 3.4 mm hole.

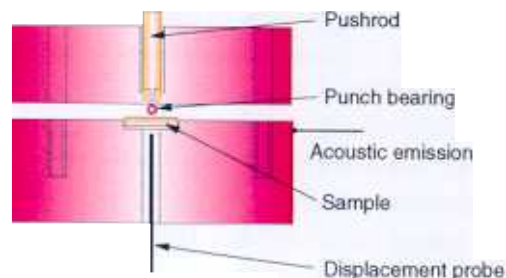


Figure1 Schematic diagram of the small punch test jig

The coated sample was placed in the rig such that the coating was strained in tension. The sample was held in place using clamping screws and the whole jig placed within a split furnace. A linear variable displacement transducer (LVDT) that had a spring loaded alumina rod extension to allow contact with the hot sample was placed at the centre of the lower surface of the sample to measure the vertical displacement.

With this arrangement tests at up to 1000 °C could be carried out. The sample was placed in the jig with both a Fecralloy® waveguide, to detect acoustic emission (AE) signals, and a thermocouple attached. It was then heated in air to the test temperature

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at $10\text{ }^{\circ}\text{C min}^{-1}$. The sample was also subject to a small servo-controlled load (30 N) during heating which compensated for system expansion during the heating cycle. Samples were then strained in about three minutes up to a maximum vertical displacement of $250\text{ }\mu\text{m}$ that amounted to a tensile strain normal to the applied load of 10%. The sample was cooled in the furnace. The tensile strain values were validated using finite element analysis. After completion of the test the samples were examined by optical or scanning electron microscopy.

Cracking of the coating was detected by monitoring AE using a waveguide that was welded to the sample. Tests were carried out with uncoated samples to determine the "background" AE signals. The Physical Acoustics Corporation supplied the AE equipment; a MISTRAS system was used which allowed a range of different AE signals to be stored. Specifically, HITS, COUNTS, ENERGY and AMPLITUDE of the signals were stored as a function of time and displacement.

Verification of the small punch test method was attempted using simple tensile tests coupled with AE where the strain to crack initiation is unambiguously measured in the tensile machine.

4 FINITE ELEMENT ANALYSIS

In the small punch test the vertical displacement was monitored by the LVDT. In order to determine the tensile strain imposed on the sample, it was necessary to convert the vertical displacement measured at the centre of the sample into tensile strain parallel to the coating. This was carried out numerically using a finite element analysis. ANSYS was used for the calculation and the input values, (Young's modulus, Poisson's ratio and yield strength) required were obtained from the literature. Calculations were carried out for the pure elastic and elastic plastic cases for room temperature and $750\text{ }^{\circ}\text{C}$.

Figure 2 shows the results of this analysis as a distribution of tensile strain in the deformed sample.

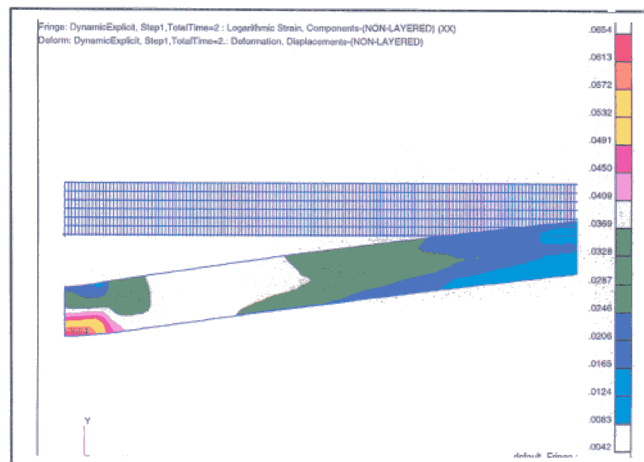


Figure 2 Finite element solution of stress distribution in a deformed SPT sample

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What is then required is to use this numerical analysis to obtain a calibration curve for the relationship between the vertical displacement at the centre of the sample and the in-plane tensile strain in the coating and this is shown in Figure 3. For a given vertical displacement above 50 μm the greater strain in the elastic/plastic compared with the elastic case is believed to be due to membrane stretching. This calibration was used for all subsequent work described below.

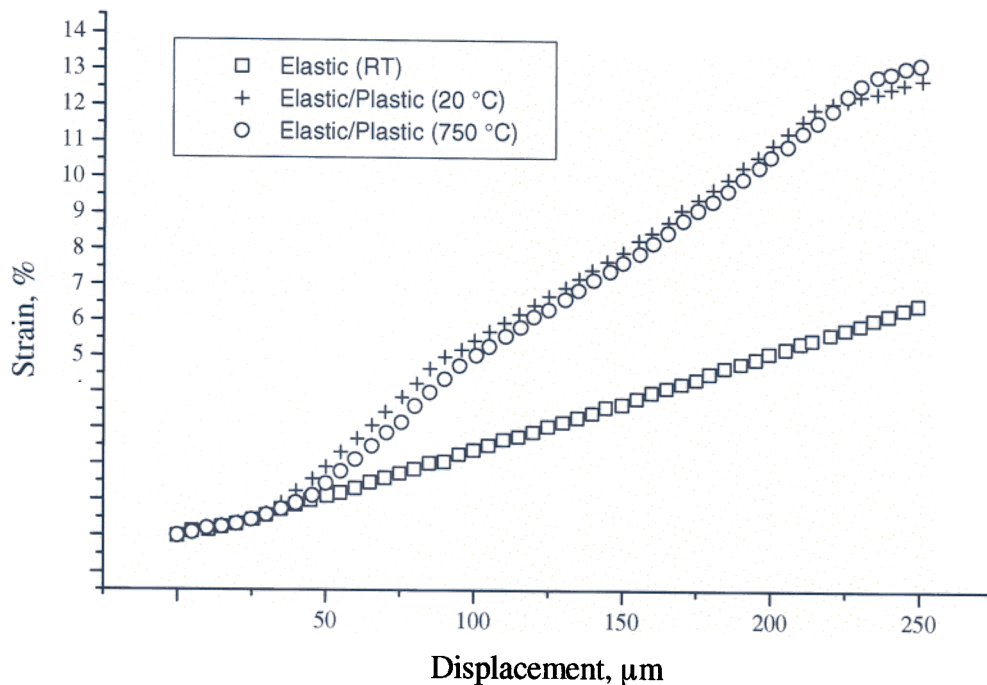


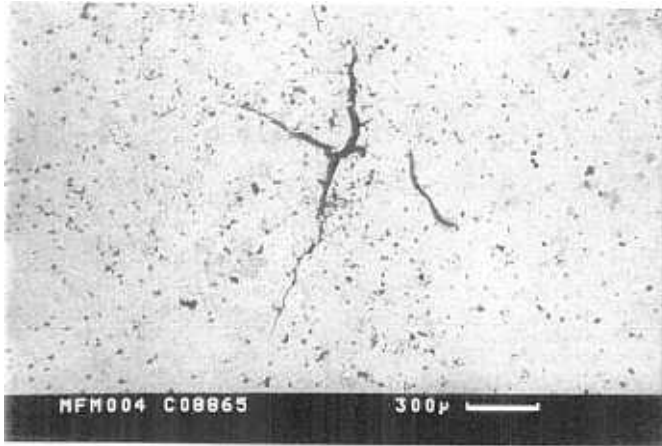
Figure 3 Calibration curve of coating in-plane tensile as a function of the vertical displacement strain in the small punch test

5 RESULTS AND DISCUSSION

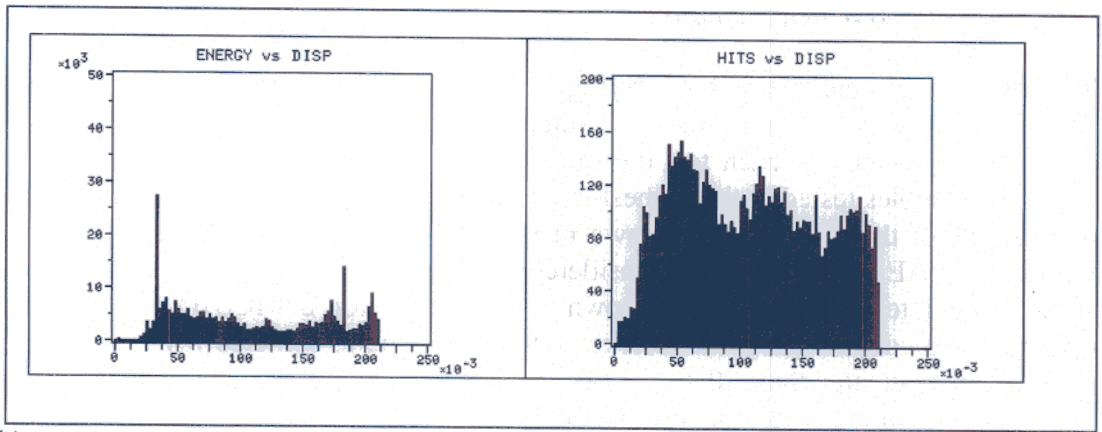
5.1 Crack detection – Optimising Acoustic Emission

During the initial experiments it was observed that while all AE signals showed a transition in behaviour during the straining cycle, it was often difficult to determine a precise time for the change in behaviour. Examination of the sample after deformation to various extents gave a good indication of the onset of cracking. Figure 4 shows a micrograph of a cracked sample and the associated AE signal (hits and energy).

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(a)



(b)

(c)

Figure 4 (a) Scanning electron micrograph a Pt aluminide coating on CMSX4 after straining to 10%, at 800 °C and the associated AE ENERGY (b) and HITS (c) as a function of vertical displacement

It was concluded from these initial experiments that the onset of the cracking could not be clearly discerned from the increase in the number a hits, but that the first high energy signals was a good indication of coating cracking. Additional tests were performed in which the same coating was strained to different extents (2.5 and 5%) and it was observed that a high AE energy signal occurred at very similar strain values (1.5 and 1.7%), giving confidence that AE energy signals were an effective indicator of cracking (Figure 5). Furthermore the AE energy values corresponding to the first cracks have similar values at about 1.6×10^4 dB. In all other experiments the first high energy AE was used to determine the onset of coating cracking.

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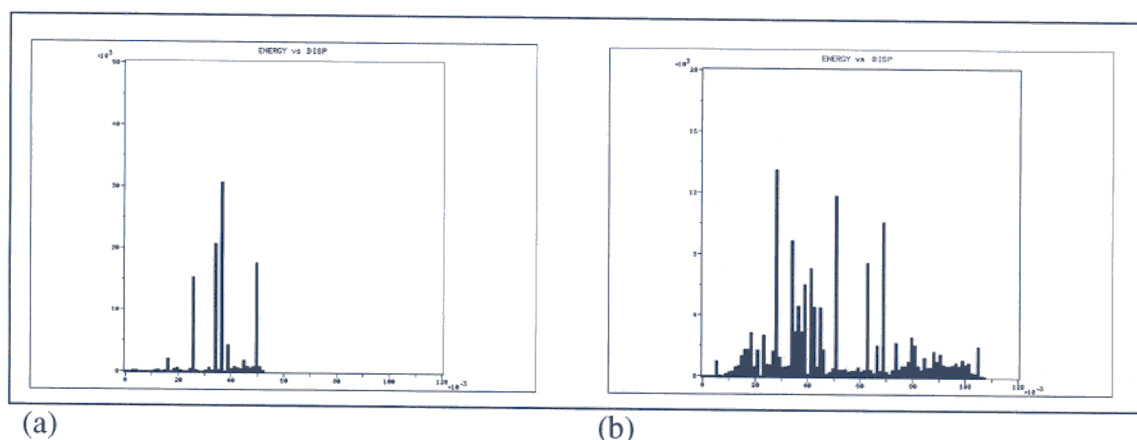


Figure 5 AE signals obtained from a Pt aluminised coating deposited on CMSX4 after testing at 750 °C strained to (a) 2.5% and (b) 5%

5.2 DBTT Curve determination

The DBTT was determined by carrying out a series of experiments with the same coating over the temperature range of interest. In most cases a strain of 10 % was applied. At the end of each test the samples were examined to check for cracking. For some samples tested at the highest temperatures ductile tearing was observed, and an example of this behaviour is shown in Figure 6. These samples did not generate high energy AE signals and are considered to be ductile. Accordingly, the fracture strain versus temperature graph shown in Figure 7a for a MCrAlY coating was constructed by determining the onset of coating cracking to coincide with the first AE energy signal greater than 20×10^3 dB. Single samples were strained generally to 10% and examined after the test to determine whether the coating had cracked. Where cracking was observed the data point is represented by a filled square and for uncracked samples the maximum strain applied is indicated by a cross. Figure 7b shows a similar plot for a typical Pt aluminide coating, and as expected the MCrAlY coating exhibited much more ductile behaviour. This approach was used for all subsequent interpretation of the data.

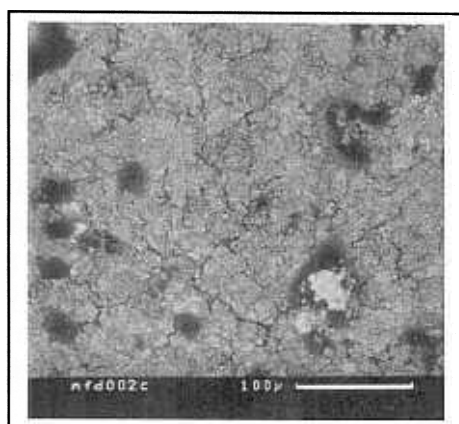
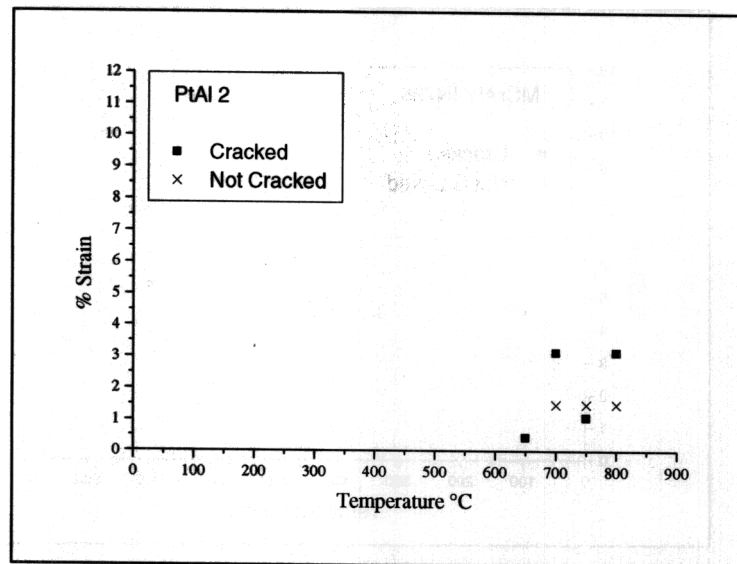
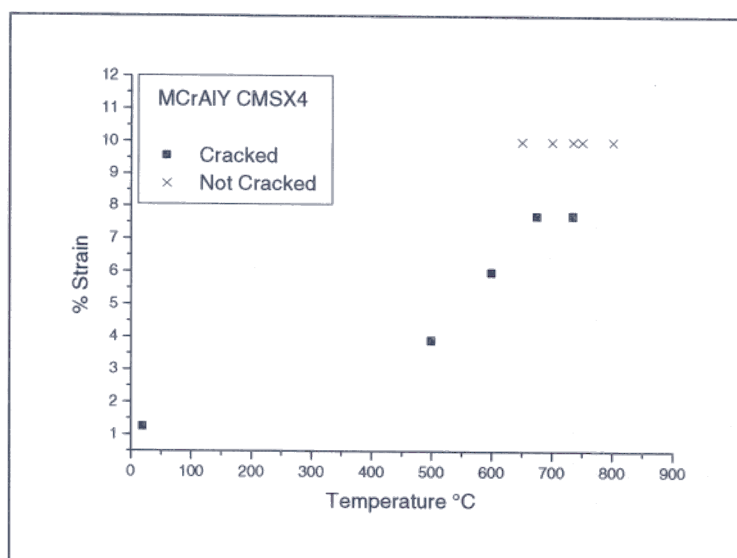


Figure 6 Scanning electron micrograph of MCrAlY (2453) strained to 10% at 800 °C showing ducting tearing.

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(a)



(b)

Figure 7 DBTT curves for (a) Pt aluminide and (b) for MCrAlY (Sicoat 2453) applied to CMSX4

The MCrAlY coating was applied to both IN738LC and CMSX4 substrates and the DBTT determined. Figure 8 shows the DBTT curve for the MCrAlY applied to IN738 and when compared with the corresponding curve for the same coating applied to CMSX4 a clear difference in behaviour is observed. The coating on the single crystal alloy, CMSX4, being more ductile. It is proposed that this difference in behaviour is a function of the different heat treatments required for the different substrates.

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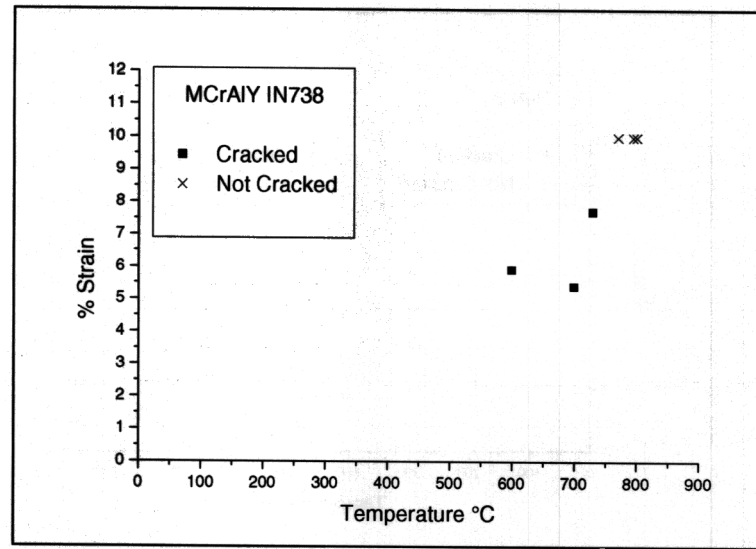


Figure 8 DBTT curve for MCrAlY (Sicoat 2453) deposited on IN738

Metallographic cross-sections were prepared through the deformed samples firstly, as added confirmation that the coatings were fractured or not as indicated by the AE energy signals, secondly, to determine whether the crack in the coating propagated into the substrate. Figure 9 shows two such micrographs and for the cases examined in this work, there were no examples of crack propagation into the substrate. For the examples shown in Figure 9 it can be clearly seen that significant plastic deformation occurred for the sample deformed at high temperature whereas very little bending of the substrate was observed in the sample tested at room temperature.

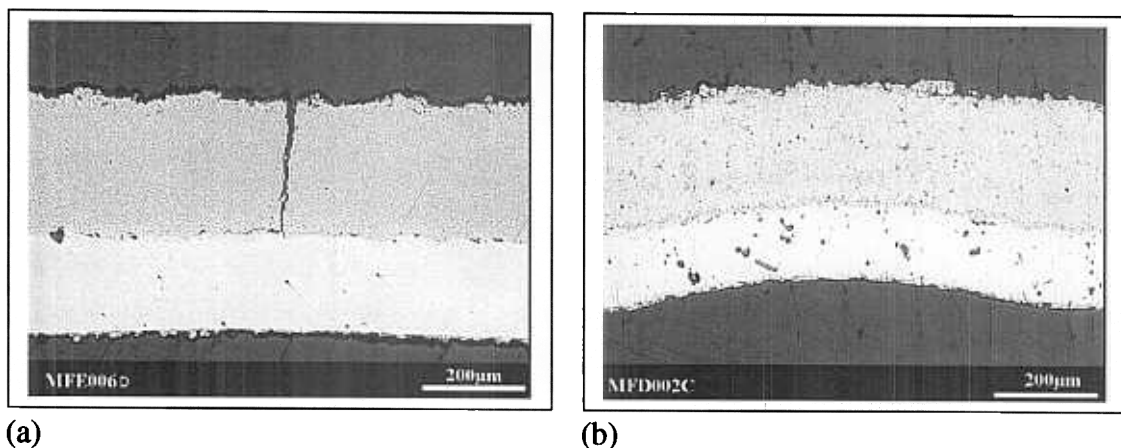


Figure 9 Optical micrographs of cross-sections through MCrAlY (Sicoat 2453) deposited on (a) CMSX4 and deformed at room temperature, and (b) IN738 and deformed at 650 °C

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5.3 Data Generation

Table I lists the results for all the systems examined during this study. The strain to cracking is determined from the AE peak energy. Generally it was observed that for the samples that cracked, the AE energy was high compared with samples that did not crack during the test.

Table I: DBTT Data for Various Coatings

<i>Substrate</i>	<i>Coating</i>	<i>Test temp, °C</i>	<i>Meas. Disp mm</i>	<i>Cracked*</i>	<i>AE Peak energy</i>	<i>Strain, %</i>
Rene80	Type 1	800	0.21	Y	12400	3.39
Rene80	Type 1	850		Y	65300	2.92
Rene80	Type 1	850	0.205	Y	33800	2.75
Rene80	Type 1	850	0.2	Y	11900	6.73
Rene80	Type 1	900	0.21	N	4218	>10
Rene80	Type 2	800	0.21	Y	53360	0.4
Rene80	Type2	700	0.21	Y	65300	4.94
Rene80	Type2	750	0.22	Y	65300	0.62
Rene80	Type 2	750	0.21	Y	26400	4.83
Rene80	Type2	800	0.21	N	22800	>10
Rene80	Type 3	800	0.21	Y	12300	3.79
Rene80	Type 3	850	0.21	Y	7077	3.67
Rene80	Type 3	850	0.21	N	3615	>10
Rene80	Type 3	900	0.21	N	13300	>10
Rene80	Type 3	900	0.205	N	300	>10
IN738	2231	600	0.21	Y	65300	5.40
IN738	2231	700	0.21	Y	21700	0.79
IN738	2231	750	0.21	Y	21700	6.56
IN738	2231	750	0.1	N	329	>5.0
IN738	RT22	20	0.26	Y	65100	0.96
IN738	2453	600	0.21	Y	65100	5.92
IN738	2453	700	0.21	Y	14400	5.40
IN738	2453	730	0.21	Y	10200	7.71
IN738	2453	770	0.21	N	3538	>10
IN738	2453	794	0.21	N	2051	>10
IN738	2453	800	0.21	N	4013	>10
CMSX4	RT22	20	0.26	Y	65600	1.48
CMSX4	RT22	801	0.2	Y	59400	4.31
CMSX4	RT22	801	0.21	Y	8051	4.08
CMSX4	RT22	820	0.22	Y	3308	0.21
CMSX4	RT22	840	0.27	Y	4244	2.64

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<i>Substrate</i>	<i>Coating</i>	<i>Test temp, °C</i>	<i>Meas. Disp mm</i>	<i>Cracked*</i>	<i>AE Peak energy</i>	<i>Strain, %</i>
CMSX4	RT22	930	0.21	Y	1297	3.39
CMSX4	PtAl 1	750	0.1	Y	13400	0.68
	PtAl 1	750	0.05	Y	30600	1.14
	PtAl 1	800	0.21	Y	18500	0.96
CMSX4	PtAl 1	800	0.1	Y	12100	0.45
CMSX4	PtAl 1	850	0.1	N	1979	>5.0
CMSX4	PtAl 1	850	0.21	Y	60000	6.56
CMSX4	PtAl2	650	0.05	Y	37000	0.45
CMSX4	PtAl2	700	0.05	N	9282	>1.5
CMSX4	PtAl2	700	0.1	Y	19200	3.15
CMSX4	PtAl2	750	0.1	Y	44400	1.08
CMSX4	PtAl2	750	0.05	N	5051	>1.5
CMSX4	PtAl2	800	0.05	N	17300	>1.5
CMSX4	PtAl2	800	0.21	Y	65300	3.15
CMSX4	2453	20	0.21	Y	65000	1.25
CMSX4	2453	500	0.21	Y	65100	3.90
CMSX4	2453	600	0.132	Y	65300	5.98
CMSX4	2453	650	0.21	N	42900	>10
CMSX4	2453	675	0.21	Y	4941	7.7
CMSX4	2453	700	0.21	N	767	>10
CMSX4	2453	750	0.21	N	1603	>10
CMSX4	none	700	0.21		4077	8.86
	none	850				

es, N = No

A criteria sometimes used to define DBTT is the temperature at which the strain to first crack is a certain level, in the Table below temperatures are listed at which the fracture strain is 1, 3 and 10%. The data are somewhat incomplete due to the limited numbers of samples that were available for this work. It is clear from this table however that MCrAlY coatings are much more ductile than the Pt aluminised systems, as is well known. Comparisons within the different types of MCrAlY show that the experimental PVD coatings were in general more brittle than the 2453 or 2231, with possibly the former exhibiting slightly more ductile behaviour. Of the three Pt aluminised coatings examined, Pt Al 2 would appear to be the most ductile, although additional work is required to confirm this. The same coating deposited on different alloys was investigated in just one case; 2453 deposited on IN738 and CMSX4. These data suggest that the coating on CMSX4 exhibited the more ductile behaviour, which may be related to differences in heat treatment for the two coating systems. It should be emphasised that in this work the coatings were tested in the as received condition, and it might be anticipated that after ageing behaviour could be modified by interdiffusion effects.

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Table II: Temperature Required for Fracture of Various Coating Systems as a Function of Imposed Strains

<i>Substrate</i>	<i>Coating</i>	<i>Temp, °C for 1% strain</i>	<i>Temp, °C for 3% strain</i>	<i>Temp, °C for 10% strain</i>
Rene 80	MCrAlY 1		800	900
Rene 80	MCrAlY 2		700	
Rene 80	MCrAlY 3		800	900
IN738	MCrAlY 2453		~500	770
IN738	MCrAlY 2231		>600	750
CMSX4	MCrAlY 2453		<500	700
CMSX4	RT22	20	930	
CMSX4	PtAl 1	750	~825	
CMSX4	PtAl 2		700	

6 SUMMARY AND CONCLUSIONS

During this work the small punch test has been developed for determination of DBTT determination of coatings. The work has demonstrated that the test can yield useful data using this relatively simple test. The test method has the advantage that it is possible to use only small amounts of material, thereby, for example, making it possible to extract test samples from turbine blades.

There have been a number of difficulties in specimen preparation that have caused erroneous data to be produced, and while the sample has a simple shape, it has been found that even small departures from the specified sample geometry, can cause serious errors. This occurs because the displacement involved in the test is very small (250 μm vertical displacement) and any error in specimen geometry or alignment of the test jig results can be a significant fraction of the overall displacement. Thus great care is needed over specimen preparation.

A further factor of concern in this work has been correlation of AE signals with cracking events. Some tensile tests were carried out on samples also studied in the small punch test. Generally good agreement was found between these two types of test giving confidence that the use of AE is valid. Furthermore, tests were also carried out in which the overall strain was reduced, to check that cracking occurred at the values indicated by the AE signals. In these cases there was good agreement with the higher strain tests.

Considering the individual DBTT curves, it is evident that the data are somewhat scattered and that it is often not possible to obtain a smooth curve. Duplicate points also frequently do not yield the same result. This could be the result of slight differences in specimen geometry between the different samples, as discussed above, but it should also be remembered that fracture is very dependent up on the presence of defects. Thus, some of the discrepancies in the results could arise from local defects in some coatings. The repeatability of the tests needs to be validated further by using

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a larger number of samples to define the DBTT curve and also by taking greater care at the specimen preparation stage to ensure uniform geometry.

7 ACKNOWLEDGEMENTS

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