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THE USE OF ACOUSTIC EMISSION TECHNOLOGY IN COATING DUCTILITY TESTING AT VARIOUS TEMPERATURES

Xin-Hai Li and Johan Moverare

Dept. of Material Technology,
Demag Delaval Industrial Turbomachinery AB,
SE-612 83 Finspång, Sweden

ABSTRACT

In this study, tests of ductility and ductile to brittle transition temperature DBTT of both PtAl RT22 and MCrAlY Amdry 997 coatings on both single crystal and polycrystalline substrates (CMSX-4, SCB, and In792) have been carried out. An acoustic emission detection technique that makes the detection of coating failures (micro cracking and delamination) possible has been employed during the tensile tests. The acoustic emission AE detection has been calibrated on the uncoated substrates and on some coated specimens at various testing temperatures and at different strain rate, together with metallurgical examination. A correlation between AE signals and failure types is established. It has been found that the substrate materials generate also some AE signals during plastic deformation. The amplitude of the AE signals depends strongly on the type of substrate material and the testing temperature but slightly on the strain rate. The substrate emissions may disturb the detection of coating failure. However, except for the disturbance from the substrate materials, the AE is still a sensitive, reliable, and useful technique to detect coating failures at various temperatures. The ductility results determined in this study have shown that the overlay coating Amdry 997 has a lower DBTT ~550°C and higher ductilities than the diffusion coating RT22. Both of these differences indicate that Amdry 997 is much more ductile than RT22.

INTRODUCTION

As gas turbine development has aggressively accelerated with increased efficiency, reliability, and availability, high temperature protective coatings applied on hot section components play an important role to allow an increase in

turbine temperature and to prolong its component life. The protective coatings used on the hot parts can usually be classified as two types, MCrAlY overlays and diffusion coatings. Due to their desired oxidation and corrosion resistance, they contain normally a much higher amount of Al and Cr elements than substrate metals do, resulting in a more brittle behaviour than that of the substrate metals. These coatings are usually applied on the first few stages of turbine components that are highly loaded, it is therefore often seen that cracks are initiated in the coatings and then propagate into the substrates causing an early failure of the components. A poor ductility of the coatings can be a limiting factor of fatigue life of the turbine components when the coatings are not properly used. Thus, the mechanical behaviour such as ductility and ductile to brittle transition temperature DBTT besides the oxidation and corrosion resistance of the coatings has become an important measure for their application ability.

When testing coating ductility together with substrates, the coatings are normally failed earlier than the substrates. Their initial coating failures, micro-cracking and delamination, can not be indicated by tensile curves due to their small thickness and moreover they are not normally visible. The initial cracks in the coatings, especially the diffusion coatings, can not be seen even using a low magnification microscope. The resistance furnaces used for high temperature testing may also keep test specimens away from the visual inspection. All these make the determination of coating ductility difficult. Many efforts have been made to determine the coating ductility by using various techniques [1-6]. One of them is a free-standing technique [2] and one removes the substrate without attacking the coating by selective etching and then tests coating alone. The drawback of this method is that its specimen preparation is too complicated

and that it can only be applied to the overlay coatings but not to the diffusion coatings since a part of the later consists of substrate metal. Acoustic emission (AE) technique has been employed to detect the coating initial failures during the mechanical testing [3-6]. The initiation of all types of coating failures such as cracking, delamination (separation from substrate) and spallation generates strong AE signals. Since the AE system can allow sampling of not only the AE signals but also the testing parameters (strain, stress, number of cycles), the coating ductility and other mechanical properties can be determined even from the testing of coated specimens. However, some substrate materials can also generate strong AE signals that can overlap with the signals from the coatings. Consequently, the substrate emissions disturb the determination of coating ductility. This problem has been observed by author's earlier investigation and mentioned in Ref. [3]. The suggestion by Ref. [3] is to use other materials that give no strong emissions. Although many publications concerning coating ductility testing using AE technique can be found in the literature database, the limitations and problems concerning the technique are seldom mentioned in the articles and no investigation on the substrate disturbance can be found in those articles. This could be the reason why the technique has not been used as a conventional testing method.

In this study, besides the coating ductility and DBTT for several coating/substrate systems, the AE response of all used substrates including single crystal materials has been carefully studied at various temperatures. The effects from the substrates on the coating ductility measurements have been investigated and discussed together with microscopic examinations.

EXPERIMENTS

Uniaxial tensile tests on both coated and uncoated specimens were performed on an INSTRON 8862 electromechanical testing machine at different temperatures. The parallel part of the test bars has a dimension of $\phi 7 \times 52\text{mm}$. Most of the tests were performed with a constant strain rate of $1 \cdot 10^{-5} \text{ s}^{-1}$. However, for some uncoated specimens the effect of strain rate on AE signal was studied by using different strain rates.

AE technique was used to detect the coating failures during the tensile tests in order to identify the strain value to coating failure. The AE behaviors of all the used substrate materials without coating were investigated in order to clarify if there is any disturbance to the AE detection of coating failures. The AE signals were detected during the uniaxial tensile testing using a system from Physical Acoustics Corporation (PAC) and analyzed by the MISTRA software. Since high temperature testing does not allow attachment of the AE sensors directly to the specimen, the sensors were applied outside of the furnace at the cooling blocks on each side of the pull rods, see Fig. 1. To avoid to much noise in the

signal a threshold value of 45 decibel was used. This value was found to shield almost all noise coming from the mechanical test machine and the cooling system but is believed to allow detection of genuine AE events coming from coating failure.

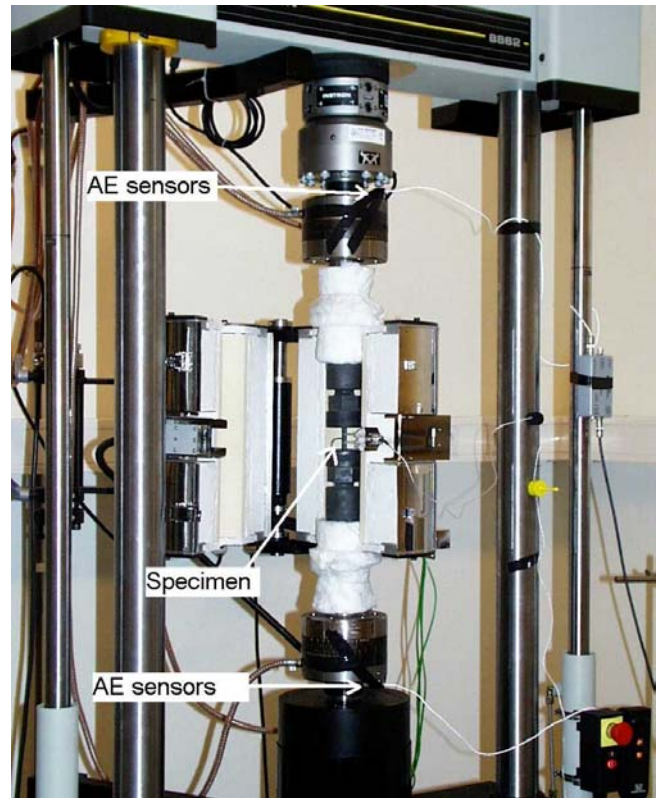


Figure 1. Experimental setup used to detect coating failure during uniaxial tensile testing.

In Table I, the coating/substrate systems tested in this study is listed together with the coating thickness. Amdry 997 is a Ni23Co20Cr8.5Al4Ta0.6Y overlay coating applied by vacuum plasma spraying, while RT22 is a Pt modified aluminide diffusion coating applied by pack cementation method. Both CMSX-4 and SCB are single crystal Ni-based alloy. The later was developed by ONERA in an EU project. The crystallographic orientations of the single crystal substrates used are also indicated in Table I. CMSX-4 test bars possess various orientations [111], [001], [011], and off-angle and therefore AE response from the uncoated CMSX-4 with different orientations was investigated to understand the orientation effects. IN792 is an equiaxed Ni-based alloy.

The coatings were post heat treated according to the solution heat treatment and aging requirements of the respective substrate materials. That is shown in Table II.

Table I. Coating/substrate combinations tested in this study.

Coating	Thickness (μm)	Substrate	Crystallography of substrate
RT22	75	CMSX-4	[111] single crystal
RT22	75	SCB	[001] single crystal
Amdry997	150	CMSX-4	Varied orientation, single crystal
Amdry997	150	SCB	[001] single crystal
Amdry 997	150	IN792	Polycrystalline

Table II. Solution and aging heat treatment requirements of the substrates.

Substrate	Solution	Aging
CMSX-4	1140°C, 2 hr, vacuum, Ar fan cooling $\leq 850^\circ\text{C}$	870°C, 20 hr in Ar
SCB	1100°C, 4 hr, vacuum, Ar fan cooling $\leq 850^\circ\text{C}$	850°C, 24 hr in Ar
IN792	1120°C, 2 hr, vacuum, cooling rate 20-40°C/min to 850°C	1 st : 845°C, 24 hr in Ar 2 nd : 760°C, 16 hr in Ar

All the tested specimens were sectioned both diametrically and axially in order to reveal the failure appearance of the coatings. The diametrical section was taken at a position of about 2/5 of the parallel length of the specimens while the axial section was made on the 3/5 part of the parallel length. Although gentle cutting was always performed, all the specimens were impregnated in Epoxy prior to the cutting and then mounted in Bakelite for later grinding and polishing. The reason for this is to easily exclude damages caused by the cutting from the analyses if there is any. An optical microscope together with an image analyzer was used for the microstructure analyses and the image analyzer was used to measure coating thickness.

RESULTS

All uncoated substrate materials were tested and their AE response all varied. Tests on uncoated specimens of IN792 show that the substrate can generate strong AE signals that may disturb the detection of coating failure. Figure 2 shows that the accumulated energy of the AE signals received from IN792 alloy in a strain range between 0.25 and 1.5% varies with testing temperature. The accumulated energy has its maximum between 700° and 750°C, but is almost not visible at temperatures lower than 500°C and higher than 850°C.

Figure 3 shows an example of the AE response from the single crystal superalloy CMSX-4. It was found that the AE signals generated by the substrate at 750°C depends strongly on the direction of the single crystal relative to the applied strain. While symmetric orientations like [001] and [111] is more or less “silent” during uniaxial tensile testing, other non symmetric orientations like [011] generate noise that is comparable in magnitude to the signals generated by the polycrystalline alloy IN792.

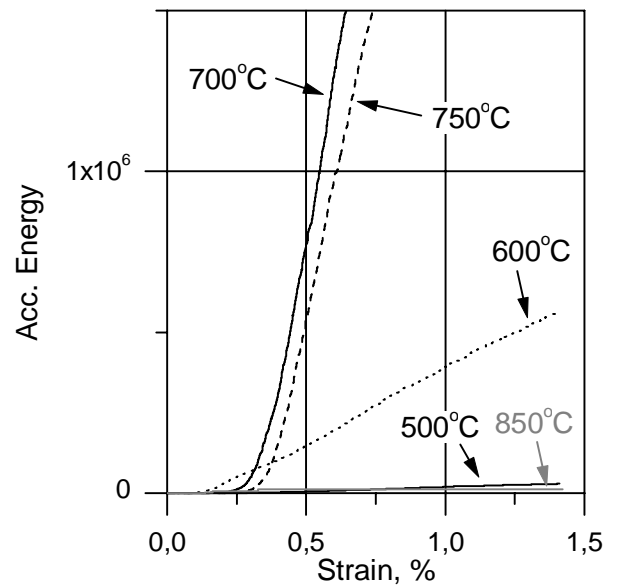


Figure 2. Effect of temperature on acoustic emission from uncoated IN792 substrate.

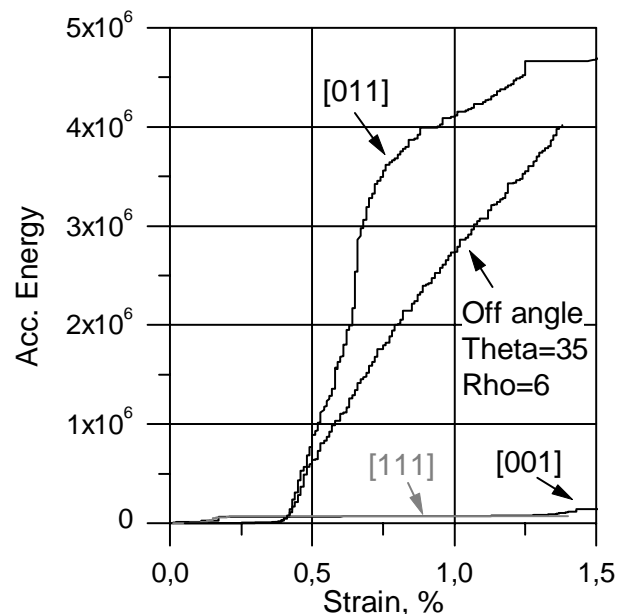


Figure 3. Effect of crystallographic orientation on acoustic emission from uncoated CMSX-4 at 750°C.

The influence of strain rate on the AE generated by the substrate has also been investigated. Two different samples of uncoated CMSX-4 [011] were tested at 750°C with different strain rates. Both a high strain rate ($5 \cdot 10^{-5} \text{ s}^{-1}$) and a low strain rate ($2 \cdot 10^{-6} \text{ s}^{-1}$) were used. It was found that the AE signals generated in the transition region from elastic to plastic deformation (0.4 -0.7% mechanical strain) is more or less independent on the strain rate, see Fig. 4(a). However, at higher strains the AE signal is continuously high for the high strain rate test but more or less vanish during the low strain rate test. At the same time as the AE response starts to differ between the two tests there is also a completely different stress-strain relationship between the two tests. While the high strain rate test shows a typical serrated flow, the low strain rate test show continues softening, see Fig. 4(b). The substrate emissions were always observed to rise at the yielding of the materials, as evidenced by Fig. 4.

The AE signal from uniaxial tensile testing of coated samples depends strongly on the type of coating/substrate system. For an overlay coating like Amdry 997, the first AE event generated by the coating is usually very distinct and indicates a “real” failure of the coating, see Fig. 5. In this study this event has been used to define the “strain to first crack” for Amdry 997. The high energy in the AE events generated by this type of coating makes it easy to distinguish from the AE noise coming from the substrate.

For a diffusion coating like RT22, the first AE event is usually not as distinct as for overlay coatings. Instead there is a gradual increase in the AE signal, see Fig. 6. This makes the definition of “strain to first crack” less obvious. It is probably the point where the energy in the AE signal significantly increases rather than the first AE event that should be associated to a “real” failure of the coating. Thus, in this case a bit more subjective determination of “strain to first crack” has to be done. In this study the point where the slope in the accumulated energy curve significantly increases has been used to define the “strain to first crack” for RT22.

While an overlay coating like Amdry 997 generates an AE signal consisting of a few AE hits with a high energy, a diffusion coating like RT22 generates more AE hits, but with a significantly lower energy. Any AE noise generated by the substrate will therefore be more difficult to separate from the AE signal coming from a diffusion coating.

The strain to failure initiation determined for the investigated coating/substrate systems are plotted for different testing temperatures in Fig. 7. It is seen from Fig. 7 that the coating ductility generally increases with increasing testing temperature, except for the Amdry 997/CMSX-4 system. The abnormal variation in ductility of the Amdry 997/CMSX-4 could be attributed to the random substrate orientation as

indicated on each of the points in the figure. The use of randomly oriented CMSX-4 was not the intention and the test bars had all mixed orientations from the supply.

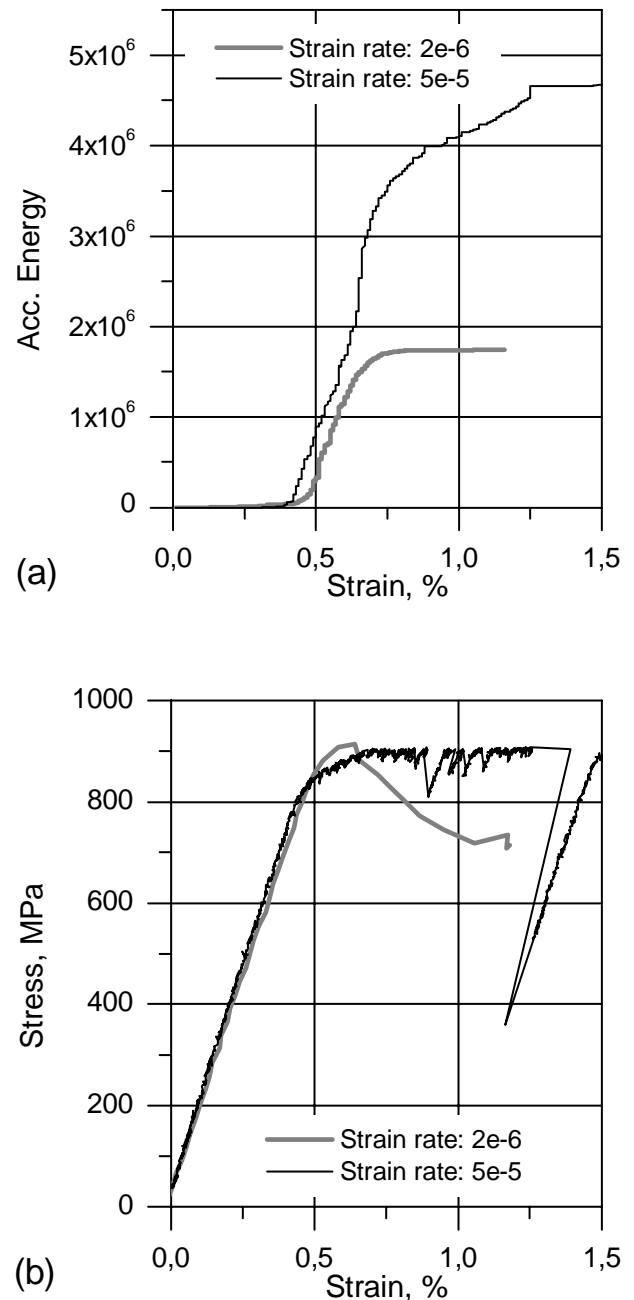


Figure 4. Effect of strain rate on (a) the acoustic emission and (b) the corresponding stress-strain relationship for uncoated CMSX-4 substrate at 750°C.

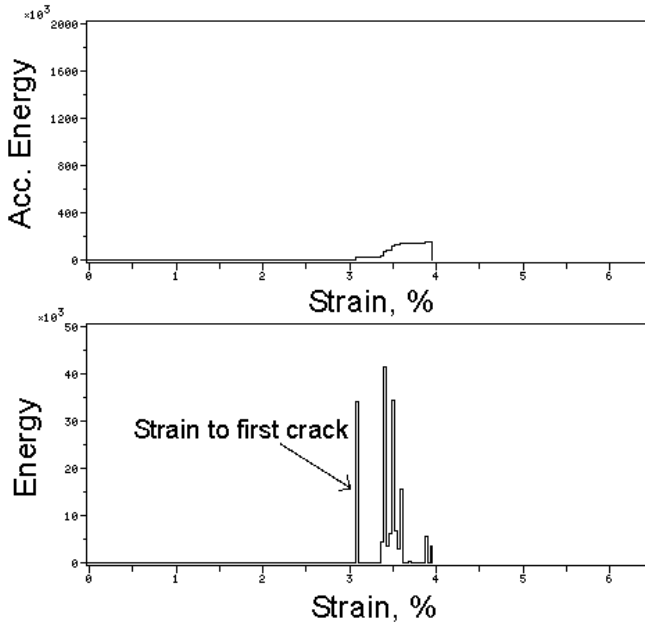


Figure 5. Acoustic emission signal for Amdry997 on CMSX-4 substrate tested at 400°C.

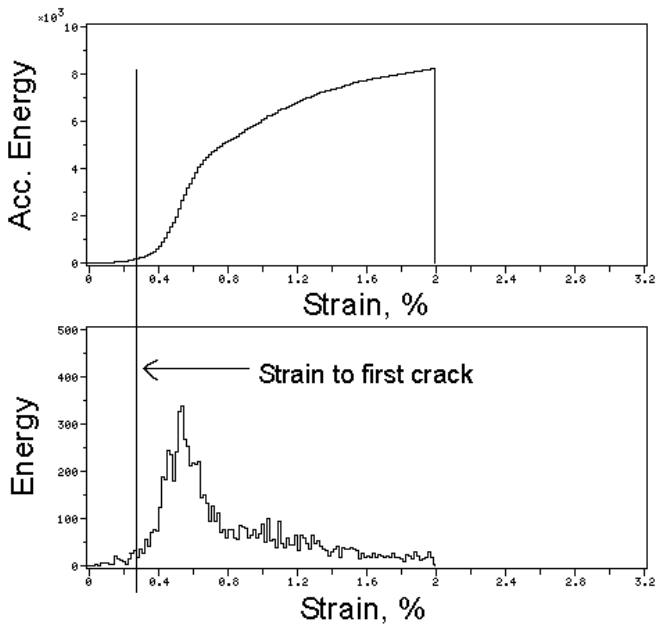


Figure 6. Acoustic emission signal for RT22 on SC2 substrate tested at 500°C.

The ductility for the rest of systems increases slowly with temperature in the low temperature range and then drastically around certain temperatures. These temperature values are defined as ductile to brittle transition temperatures, DBTTs for coatings. According to the figure, RT22 has a DBTT between 700 and 750°C while Amdry 997 has a lower one between 500 and 600°C. Moreover, Amdry 997 has always a much higher

ductility value than that RT22 has at each temperature. These great differences all indicate that Amdry 997 is much more ductile than RT22. Besides the differences between the two coatings, the coating ductility and DBTT were also influenced by the substrate. For the diffusion coating like RT22, this is obvious due to the fact that the coating is a product of the interdiffusion between the coating elements such as Al and Pt and the substrate elements and the properties of the coatings should depend also on the substrate under.

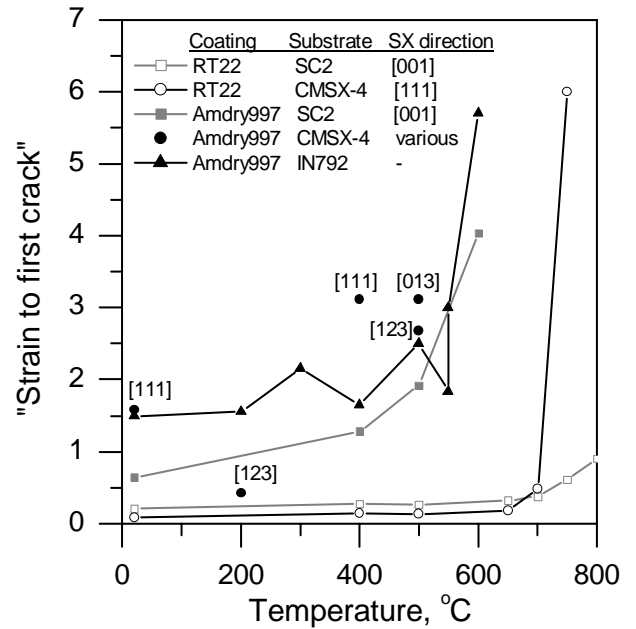


Figure 7. Coating ductility at various temperatures

Appearances of coating failures in the tested specimens were analyzed by microstructural examinations. It was found that the failure appeared differently, depending on type of coating, substrate, and testing temperature.

Figure 8 is a micrograph taken on an axial section of 700°C tested RT22 on CMSX-4, showing a typical crack appearance in the tensile tested RT22. It should be noted that the crack was opened up by a plastic deformation of the substrate since the test stopped at 0.9% in the plastic strain range (see Fig. 3.) while the crack initiation occurred at 0.5% strain. The crack is circumferential and the cracking is the only failure mode observed in RT22 no matter what substrate and testing temperature is used.

In contrast to RT22, both cracking and delamination were observed in the Amdry 997 overlay coating. Figure 9 is a micrograph taken from an axial section of the Amdry 997 coated CMSX-4 specimen tested up to 2.75 % strain at room temperature, showing only one of the cracks initiated at 1.57% strain. In this specimen, the coating failed with only cracking.

However, in some specimens both coating delamination at coating/substrate interface and cracking were observed. Figure 10 displays an example of the failures observed in an Amdry 997/IN792 specimen tested up to 4% strain and at 500°C. In the figure the separation (delamination) between the coating and the substrate is obvious.

Table III summarizes the failure types observed from the microscopic examination, testing temperature, measured thickness, cracking strain, and ending strain for all tested Amdry 997/substrate combinations. It is seen in the table that the coating failed with only cracking during the room temperature testing except for the Amdry 997/SCB combination. The delamination in combination with big cracks mainly occurred in the intermediate temperatures 200-500°C. When the testing temperature was increasing to the higher temperatures around the coating DBTT point (~550°C see also in Fig. 7) or above, again only the fine coating cracks or specimen fracture were observed. The delamination is a result of a radial tensile stress between the coating and the substrate (delamination stress) induced by a radial substrate contraction. In the intermediate temperature range the coating was still brittle or rigid and did not follow the substrate contraction so that the delamination stress was high and the delamination occurred. When the temperature reached DBTT or above, the coating became ductile and then could follow the substrate contraction, therefore the delamination stress was too low to lift up the coating. The substrate contraction caused also the initiation of the vertical cracks in the coating from the coating/substrate interface as seen in Fig. 11.

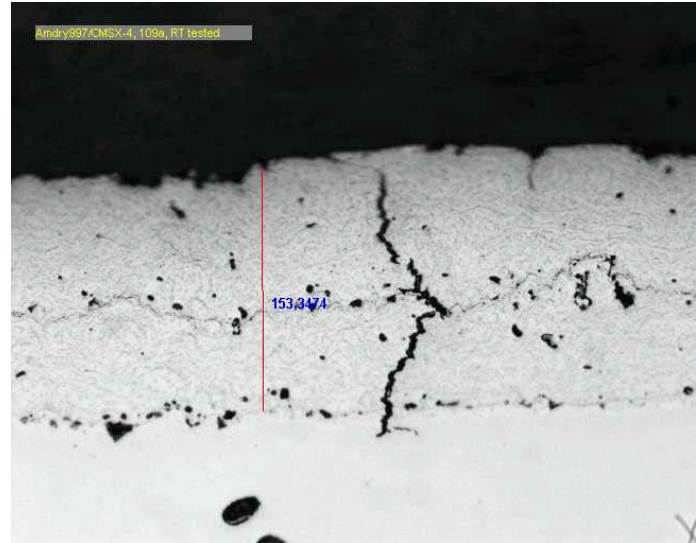


Figure 9. Crack morphology in Amdry 997 coated on [111] CMSX-4 tested at 700°C.

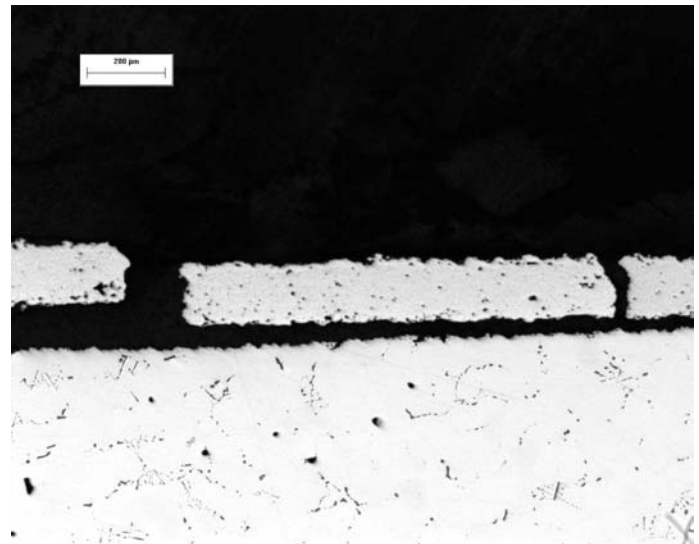


Figure 10. Delamination and big cracks observed in an Amdry 997/IN792 specimen tested up to 4% strain and at 500°C.

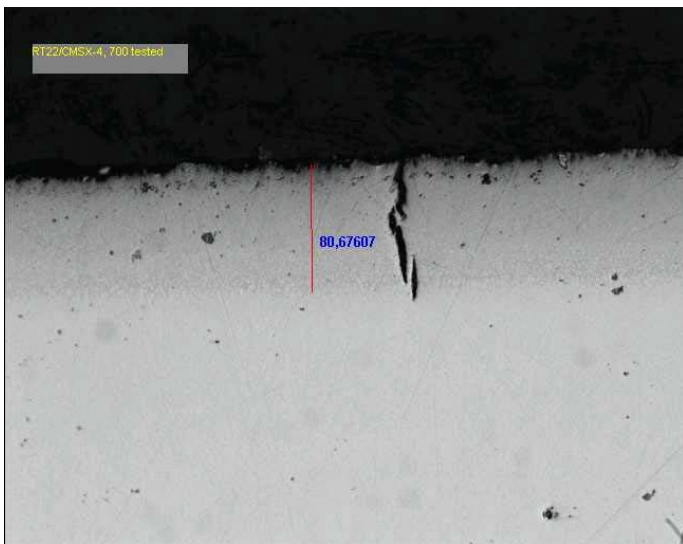


Figure 8. Crack morphology in RT22 coated on [111] CMSX-4 tested at 700°C and up to 0.9% strain.

The big cracks in the coating were always observed together with delamination and the AE signals received during the coating failure can be generated from both the delamination and cracking. This may explain why the AE signals appear one by one separately (Fig. 5) unlike the ones from RT22 that pop up as a broad peak (Fig. 6).

Table III. Failure observation in all Amdry 997/substrate specimens and material and testing information is also summarized.

Base material	Orientation	Test temp	Failure observation	Ending strain	Failure strain	Coating thickness (microns)
CMSX-4	111	22	Fine cracks	2,75%	1,57%	148
CMSX-4	123	200	Big cracks+delamination	2%	0,42%	147
CMSX-4	111	400	No visible cracks	4%	3,11%	161
CMSX-4	123	500	Big cracks+delamination	2,75%	2,68%	139
CMSX-4	013	500	Small+short cracks	3,90%	3,11%	147
SCB	001	22	Big cracks+delamination	1,15%	0,62%	140
SCB	001	400	Big cracks+delamination+substrate cracks	2,10%	1,28%	150
SCB	001	500	Big cracks+delamination	2,20%	1,78%	144
SCB	001	600	Specimen fracture but no visible cracks	4%	>4%	144
IN792	Poly	22	1 crack	2%	1,50%	150
IN792	Poly	200	delamination+cracks	2,10%	1,55%	140
IN792	Poly	300	delamination+cracks	2,80%	2,15%	150
IN792	Poly	400	delamination+cracks	1,80%	1,65%	143
IN792	Poly	500	delamination+cracks+substrate cracks	4,00%	2,50%	150
IN792	Poly	550	cracks	3,60%	3,00%	149
IN792	Poly	600	cracks	6%	5,70%	145

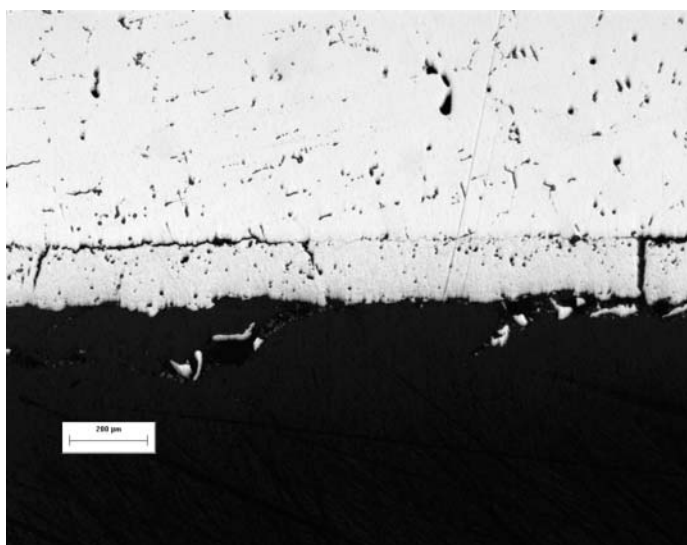


Figure 11. Crack initiation from coating/substrate interface caused by substrate contraction.

In Table III, it is also seen that the measured coating thickness has a relatively small variation around the desired value 150 μm shown in Table I. For the CMSX-4 substrates with various orientations, the correlation between the large variation in failure strain and the failure type can be seen, i.e., the fine cracks are related to the large failure strains while the delamination and big cracks are connected to the small failure strain values.

It should be noted that the Amdry 997 coating tested contains a amount of pores and inclusions at the coating/substrate interface as seen in Figs 9 and 11. This should be responsible for a weak bonding at the interface and therefore a low delamination strain.

Since the failures in Amdry 997 were caused by different loading direction, i.e., either by axial tensile stress or a combination of both axial and radial tensile stresses, one has to take into account the differences in failure types and in substrates when evaluating and comparing the failure strains.

As shown in Table III, no visible crack was observed in the axial section of the Amdry 997/[111] CMSX-4 tested at 400°C up to 4% strain. The reason to miss the cracks could be that there were only a few cracks and they located on the part where the diametrical cross-section was taken.

DISCUSSIONS

In this study AE technology has been used successfully to detect the coating failure and determine the coating ductility in most of the cases, however one should pay attention to the substrates. The substrates may affect the determination of coating ductility. There are two major substrate effects observed in this study:

1. Strong AE signals received from polycrystalline IN792 and [011] and other off-angle single crystal CMSX-4 in a

temperature range 600-750°C overlaps the signal from the coating failures and disturb the measurements, as seen in Fig. 2 & 3.

2. Different orientations of CMSX-4 lead to a big variation on failure type and failure strain measured, as seen in Fig. 7. But this effect can only disturb the measurement of the overlay coating like Amdry 997.

For the 1st substrate effect, the AE signals received from the substrates and already from the yielding of the materials are generated during the plastic deformation [7]. They are resulted from some abrupt and permanent changes in the materials, e.g., dislocation movement. The high amplitude of the signals can be due to high strain rate, cast structure, thick section, large grain size, brittle deformation, high barrier to dislocation movement, low testing temperature, and etc. [7, 8]. Our results of the strain rate study (Fig. 4) confirm the strain rate effect. However the relatively low strain rate that we have tested can not eliminate the strong substrate emissions. Our finding of the strong substrate emission is limited in a temperature range between 600-750°C and can not be easily explained by the above factors. One possible explanation to this is that just within this temperature range the cast Ni-superalloys studied here has experienced a brittle deformation and a strong interaction between precipitates and dislocations. In case of the different AE responses at different crystallographic orientations of single crystal CMSX-4, the explanation can be that the obstacle (barrier height) to the dislocation movement differs with the orientation.

The substrate emissions that only appears in the temperature range 600-750°C however do not disturb the measurement on the Amdry 997 type of overlay coatings since such coatings normally have sharp and high AE peaks and also a DBTT not more than 600°C (at this temperature the AE signals from the substrates are weak and do not cover the coating signals). Thus, to our understanding it is not necessary to use a ductile substrate material for the overlay coatings as suggested by Ref. [3]. For the diffusion coatings like RT22 that have normally a DBTT just in the 700-750°C temperature range, the substrate disturbance is really a hinder for using AE to determine DBTT, but the determination of the coating ductility at the temperatures below 600°C is still feasible.

The 2nd substrate effect is believed to be due to the different degree of the radial contraction of CMSX-4 at different orientation. The specimens on the [111] substrate that have much higher ductility values than those on the other orientations have only fine cracks while the others have both lower ductility values and coating delaminations. This indicates that the [111] substrate has the smallest radial contraction among the others. It is understandable that the poisson's ratio depends on the crystallographic orientation and

so does the substrate contraction. To avoid the orientation effect, one should choose the same orientation of the same single crystal substrate when the AE technology is used. The orientation effect could also affect the testing on the polycrystalline substrate when it has different preferential orientations. This may explain why the data points of Amdry 997/IN792 jump somewhat up and down as seen in Fig. 7.

From the results in Table III and above discussion, it is evident that the use of [111] CMSX-4 can result in the least radial contraction and therefore no delamination up to a relatively high strain level and moreover their failure strain are much higher than those with other orientations. In this case a true coating failure strain (cracking strain) can be determined. Manufacturing of such test specimens is now in progress and the tests will be performed within the project frame in order to further prove it.

This can also be true to the other Amdry 997/substrate systems. Unlike the RT22 diffusion coating, the overlay coating Amdry 997 is just an added layer on the substrates and it's properties should not depend strongly on the substrate used. However, the failure strains of the coating in Table III and Fig. 7 shows a big difference between different substrates. This is mainly because of the delamination and different degree of substrate contraction. Making a comparison between all room temperature tested specimens (marked with gray) and a comparison of the 400°C tested ones (marked with dots), it could be seen that the lower failure strains are always resulted from the delamination. If a substrate with a small radial contraction is used the failure strain may not so much dependent on the substrate.

CONCLUSIONS

1) Amdry 997 has a higher failure strain at all testing temperatures and a lower DBTT than RT22 does. The DBTT of Amdry 997 is ~550°C while that of RT22 is ~750°C. Both of these differences indicate that Amdry 997 is much more ductile than RT22.

2) RT22 has failed only with cracking perpendicular to its surface, while Amdry 997 has failed with cracking or both cracking and delamination depending on testing temperature, type of substrate, and substrate orientation. The coating delamination is due to the radial contraction of the substrate.

3) Two substrate effects have been found to affect the coating testing carried out in this study:

The 1st effect is the strong AE emissions from some substrates in the temperature range 600-750°C and the amplitude of the substrate emissions from the single crystal substrates depends on their crystallographic orientation.

The 2nd effect is that the substrate contraction of single crystal substrate is dependent on the orientation, leading to a large variation of the coating failure strains. It is suggested that the same orientation should be chosen for this type of testing.

4) A low strain rate reduces the amplitude of the AE signals received from substrates.

5) The substrate emissions occurred in the temperature range 600-750°C only disturb the determination of DBTT point of the diffusion coatings at least in this study.

ACKNOWLEDGMENTS

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