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Increase in ductility of Pt-modified nickel aluminide coating with high temperature ageing

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ABSTRACT

The ductility of β -(Ni,Pt)Al coating on the single-crystal nickel-base superalloy AM1 has been studied after isothermal and cyclic ageing at 1100 °C. Using accurate in-situ surface observations during tensile tests at room temperature and subsequent digital image correlation, the critical local strain required for crack initiation in the coating was obtained. Such an analysis is essential in order to directly identify the cracking of the β -(Ni,Pt)Al coating during tensile tests and to understand the mechanisms responsible for the evolution of coating ductility induced by thermal ageing. It is shown that both isothermal and cyclic ageing lead to increases in critical local strain, as compared to unaged sample. Besides, the sample after cyclic ageing possessed a greater number of cracks for similar ageing durations as those used for isothermal conditions. However, after the longest ageing duration the coating was either not cracked at all (cyclic ageing) or cracks were not observed in-situ by optical microscopy and only identified afterwards by scanning electron microscopy due to their small dimensions. These observations have been discussed using experimental surface profile analysis and microstructural observations combined with finite element analysis. The results suggest that the surface roughness was responsible for the coating cracking for the short ageing durations (when γ' just starts to appear in the coating) and for lengthy ageing (with increased γ' phase fraction) the γ' phase ensured coating ductility.

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1. Introduction

Nickel aluminide coatings are widely used to increase the corrosion and oxidation resistance of nickel-base superalloys. At high temperature in air the aluminium provided by the coating enables the formation of α -Al₂O₃ thermally grown oxide (TGO) which then protects the superalloy from further oxidation [1,2]. Pt-modified nickel aluminide coatings are replacing plain nickel aluminides; Pt increases TGO adherence [3–5] as well as reduces oxidation kinetics. However, the coating deposition on nickel-base superalloys usually leads to undesirable reductions in mechanical properties of the coating/substrate system [6–11]. Generally, such a degradation is attributed to poor mechanical properties of the coating of a β -NiAl intermetallic phase, as compared to the superalloy substrate [10,11,51].

Over the last few decades special attention has been paid to the evolution of the mechanical properties of coating/substrate systems during thermal cycling in atmospheric air, since this is close to service conditions. For example, it has been shown that cyclic strains in the plain β -NiAl coating induced by thermal cycling were able to generate coating cracking and even crack propagation into the superalloy substrate [12].

It is now well established that changes in the coating microstructure during thermal cycling have an important effect on the mechanical properties of the coating. Totemeier et al. studied [13] the importance of the microstructure of the β -NiAl coating on the coating fracture strain: samples for tensile testing pre-exposed at 850 °C and at 1100 °C for 140 h exhibited significantly different coating fracture strains (0.57 and higher than 5%, correspondingly), since the former still possessed an untransformed β -NiAl coating, whereas for the latter the coating had transformed into ductile γ' with small subsurface β grains remaining. Crack initiation for the specimens pre-exposed at 850 °C was always observed to occur on the surface, whilst for those pre-exposed at 1100 °C the crack initiation was observed in subsurface β grains or at subsurface β/γ' interfaces. In the case of untransformed coatings the crack path mainly was intergranular, although some transgranular cracks were

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observed. Moreover, crack propagation was arrested by the coatingsubstrate interfaces. The same observation was made during thermo-mechanical fatigue (TMF) degradation of β -NiAl [14] and β -(Ni,Pt)Al [9] coatings on single-crystal nickel-base alloys. In addition it was observed that, during the initial stages of $\beta \rightarrow \gamma'$ phase transformation which occurred in the coating during cyclic ageing in air, through-thickness cracking along the β/γ' interface could develop in the coating during tensile tests and lead to a significant reduction in ductility of the coating/substrate system [10].

The evolution of the microstructure of the coating during thermal cycling as well as in TMF experiments was always accompanied by an important increase in surface roughness, so-called rumpling, that developed for both plain and Pt-modified nickel aluminide coatings [12,15–22]. The role of surface roughening in initiating cracking of the coating remains questionable. Crack initiation at surface defects that represented slightly roughened and depressed areas relative to the rest of the coating was observed in Refs. [13], but it was not possible to conclude whether such surface defects were due to the roughness developed during thermal ageing or to the coating deposition process. The interaction of rumpling and oxidation was suggested as favouring crack propagation into the superalloy substrate during sustained peak low-cycle fatigue [23]. Therefore, the influence of rumpling on coating ductility needs to be examined more in detail. Moreover, a recent study of oxide spallation indicated the critical role of the surface roughness for strain localisation [24].

It is worth noting that the conclusion about the evolution of mechanical properties of the coating/substrate system due to thermal ageing is based on results of mechanical tests of the coated samples mainly analysed at a macroscopic scale. It seems however that such an analysis hinders real coating properties, since the cracking of the coating can take place long before coating/substrate failure due to the brittleness of the β -(Ni,Pt)Al phase. In other words, the superalloy substrate can continue to deform plastically with an already cracked coating. The cracked coating does not protect the superalloy from oxidation, since it can take place in the cracked regions. It is important therefore to be able to evaluate coating ductility independently of the superalloy ductility which dominates during tensile tests. Recent studies of the mechanical properties of free-standing Pt-modified nickel aluminide coatings using microtensile tensile samples have suppressed the influence of the substrate ductility [10,25,26]. However, thermal ageing of such free-standing coatings cannot induce changes in the microstructure of the coating observed in service conditions, since the superalloy substrate acts as a source of different elements during the diffusion activated by thermal ageing. Therefore, the influence of thermal ageing on coating properties should be studied preferably on the complete coating/substrate system.

A special experimental procedure was therefore used in this work to evaluate both β -(Ni,Pt)Al coating ductility as well as to investigate the role of rumpling on coating ductility after thermal ageing: in-situ observations of the coating surface were made during tensile tests up to first crack appearance in the samples after different ageing conditions (isothermal and cyclic) and durations. Subsequently digital image correlation was used to evaluate the local critical deformation (i.e. deformation required for appearance of the first crack).

As mentioned earlier, such an accurate analysis is essential in order to understand the mechanisms responsible for the evolution of the coating ductility induced by thermal ageing. Such knowledge is required for the development of new materials with enhanced oxidation resistance for the aeronautical industry. The competition between the effects of the surface and of the microstructure leading to cracking of the coating or its ductility, as it is discussed further, is of fundamental interest, since it can be applied to any type of coated materials.

2. Experimental

2.1. Material

The chemical composition of the nickel-base single crystal superalloy AM1 used in the present work is shown in the Table 1.

The deposition of Pt-modified nickel aluminide coating on the samples for tensile tests consisted of several stages: (i) Pt electrodeposition (final Pt layer thickness of $6-8 \mu m$), followed by (ii) diffusion treatment under vacuum at 1050 °C for 90 min, and, finally, (iii) Al phase vapour deposition (APVS: Aluminizing Phase Vapour Snecma) [27]. After the described procedure the coating consisted of a β -(Ni,Pt)Al phase with a thickness of approximately 40 µm; the interdiffusion zone (IDZ) was formed between the coating and the superalloy substrate and contained β , γ' phases and so-called topologically close packed phases (TCP) (Fig. 1a). A line of alumina grit particles was observed as well. These alumina particles were introduced during the grit blasting process used to increase the surface roughness of the superalloy substrate so as to increase coating adherence. Initial Root Mean Square (RMS) roughness of the surface was about $0.9 \pm 0.4 \,\mu m$ (Fig. 1b) (see Section 2.5 for the details of microstructure analysis).

2.2. Sample geometry

The tensile samples used for the thermal ageing and further mechanical tests were optimised based on a specific geometry designed previously [28]. The long axis of the sample coincided with the [001] direction of the single-crystal substrate within deviations of $8-9^{\circ}$. The samples had a total length of 66 mm; within the gauge length the samples had a homogeneous thickness of 2 mm and the width varied from 8 mm (at the gauge centre) to 12 mm (at the gauge edges) (Fig. 2).

2.3. Thermal ageing

The ageing was carried out in air under both isothermal conditions at 1100 °C and thermal cycling. Each thermal cycle lasted approximately for 1 h; it included heating from 100 to 1100 °C, soaking during 45 min and, finally, cooling to room temperature. The number of applied cycles was equal to N_{min} , N and N_{max} (where $N_{min}/N_{max} = 0.25$, $N/N_{max} = 0.5$) and thus the time cumulated at 1100 °C was $0.75N_{min}$, 0.75N and $0.75N_{max}$ hours, respectively. Under the isothermal conditions the holding time (in hours) was equal to N_{min} , N and N_{max} . During such high temperature ageing in air, an α -Al₂O₃ TGO layer was formed on the surface of the sample due to oxidation (Fig. 3).

2.4. Tensile tests

After thermal ageing, as described above, the mechanical tensile testing was performed so as to induce cracking of the sample and, thus, to enable the in-situ measurement of the critical macrostrains by a contact extensioneter and defined as the minimum global

Table 1

Chemical composition of the nickel-base superalloy AM1 used in the present study (wt. %).

| | Со | Cr | Мо | W | Ta | Al | Ti | Ni |
|------|----|----|-----|---|-----|-----|-----|------|
| Min. | 6 | 7 | 1.8 | 5 | 7.5 | 5.1 | 1 | Bal. |
| Max. | 7 | 8 | 2.2 | 6 | 8.5 | 5.5 | 1.4 | Bal |

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