SOLID STATE INTERFACIAL REACTIONS IN A CERIA-COATED Ni-BASED SUPERALLOY

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ABSTRACT

Perovskite type phases are known to improve the adherence of protective oxide scales grown onto chromia- and alumina-forming alloys and coatings. The present study investigates the interfacial reactions occurring during the high temperature oxidation of a ceria coated Ni-based superalloy at 1100°C. The substrate firstly grew a duplex oxide scale with a NiAl_2O_4 spinel on top of an $\alpha\text{-Al}_2\text{O}_3$ scale underneath the ceria coating. Increasing oxidation time led to the development of Al-rich and Ce-rich nanometric grains at the oxide scale/coating interface and to the progressive vanishing of the NiAl_2O_4 spinel. Micro-Raman analyses demonstrated the formation of cerium aluminate CeAlO_3 by solid-state reaction between Al_2O_3 and CeO_2 favoured by an Al supply coming from the substrate. The results are in line with the thermodynamic calculations.

KEYWORDS: Ceria; Alumina; Perovskite; Solid-State Reaction; Oxidation; Coating.

1.- INTRODUCTION

The beneficial effect of the addition of rare-earth elements on the high temperature oxidation behaviour of chromia- and alumina-forming alloys and/or coatings has been widely reported and discussed in the literature [1-7]. Commonly agreed effects are that the addition of reactive elements alters the transformation kinetics of transient aluminas generally observed during early stages of oxidation and changes in the growing mechanisms of the oxide scale [1-7]. These modifications may lead to slower oxidation kinetics [8-10], reduced diffusion processes

[1,8,9,10], modification of the morphology and improvement the adherence of the oxide scales [11-17]. These phenomena have been ascribed to the segregation of reactive elements as single or mixed oxide at the oxide grain boundaries and at the metal/oxide interface [18-20]. Further, the formation of perovskite compounds is known to improve the adherence of protective oxide scales onto oxide-coated alumina-forming alloys [21,22]. Growth of (RE)AlO₃ perovskite phases by solid state reactions at high temperature has been demonstrated for Y₂O₃- and Nd₂O₃-coated [21] and Y₂O₃-ODS [23] alumina-forming substrates. The perovskites improved the adherence of the oxide scale formed and blocked diffusion of the ionic species, hence resulting in overall improved oxidation resistance [24,25]. In some of our previous studies, the formation of CeAlO₃ at the alumina/ceria interface was also considered to contribute to the enhanced adherence of the oxide scale of electrolytic CeO₂ coatings on Ni-based superalloy substrates under isothermal and cyclic conditions [26-28] even after the application of water drops to embrittle the oxide/coating interface [29,30]. Nevertheless, despite favourable thermodynamic conditions, this was not clearly demonstrated experimentally.

In the open literature, few studies deal with growth of perovskites by reaction between CeO₂ and α -Al₂O₃ under oxidative atmosphere. Indeed, most of the investigations employed inert or reducing atmospheres to reduce Ce⁴⁺ and to favour the formation of the phase CeAlO₃ by reacting with transition aluminas at high temperatures for catalytic applications [31-34]. Otherwise, synthesis of CeAlO₃ by arc melting [35] and through wet methods and calcination of powders [36,37] have also been reported. Yet, it is worth mentioning that such experiments were carried out using free-standing powders precursors. In the particular case of CeO₂-based coatings deposited onto a metallic substrate, solid state reactions will not only depend on the temperature and treatment duration and atmosphere but also strongly on the underlying substrate chemistry.

Therefore, the present study aims at understanding interfacial reactions during the oxidation of a ceria-coated Ni-based superalloy using Raman spectroscopy to emphasize the likely formation of mixed oxides supported by thermodynamic calculations.

2.- MATERIALS AND METHODS

Coupons (12 mm diameter, 2 mm thick) of grit blasted (220 mesh alumina) single-crystal René N5 Ni-based superalloy (composition given in Table 1) substrates were electrolytically coated with Ce hydroxides using a three-electrode set-up for 20 min following the experimental conditions given in [26]. The coatings were subsequently annealed in flowing argon atmosphere

at 1050°C for 1h to enhance adherence and provide pre-oxidation of the substrate [38]. The ceria-coated superalloy substrates were then submitted to cyclic and isothermal oxidation at 1100°C in order to study the gas/solid heterogeneous oxidation mechanisms and the solid-state interfacial reactions.

Table 1.- Nominal composition (wt%) of the René N5 Ni-based superalloy of this work.

Ni	Cr	Co	Mo	W	Ta	Re	Al	Hf
Bal.	7	8	2	5	7	3	6.2	0.2

The samples were characterized by X-ray diffraction in the Bragg-Brentano mode between 10 and 90° (step size of 0.02° for 2 s and slit sizes of 0.6 mm) using a Bruker AXS D8 Advance (λ_{Cu}) and the surface and cross-sections were subsequently studied by Scanning Electron Microscopy coupled to EDS analyses (SEM/EDS, FEI Quanta 200F/EDAX). Likewise, local micro-Raman analyses were performed at room temperature to raster surfaces and cross-sections with a Jobin Yvon Horibat LabRam HR Raman spectrometer equipped with a microscope and a Peltier-cooled CCD detector. The spectra were recorded with the acquisition software LabSpec with a resolution of $\sim 0.2 \text{ cm}^{-1}$. Excitation was provided by a He-Ne laser source (632.8 nm). The laser power has been chosen in order to avoid local heating of the samples and provide sufficient good resolution [39]. The spot under the x50 objective was set to have a diameter lower than 3 μm .

The thermal events were studied with a SETARAM LabSys Evo TGA-DSC device by heating powder mixtures of CeO_2 ($< 5 \mu\text{m}$, 99.9% Sigma-Aldrich), Al_2O_3 (99.997%, Sigma-Aldrich) and Al (5-6 μm 99.99%, Sibthermochim) at $10^\circ\text{C}.\text{min}^{-1}$ from 40°C to 1500°C under flowing argon gas (20 $\text{mL}.\text{min}^{-1}$) to limit oxidation. Thermodynamic calculations were also made using the HSC Chemistry thermodynamic software [40].

3.- RESULTS AND DISCUSSION

3.1- As-coated condition

The cross-section of the ceria-coated Ni-based superalloy substrate after thermal treatment at 1050°C in flowing argon atmosphere for 1h is shown in Fig. 1 (a). The ceria coating exhibits a foam-like grainy microstructure with a micro-cracks network, some being open and leading directly to the coating/oxide scale interface. The full crystallization into the stoichiometric CeO_2 fluorite form is also promoted, as suggested by the narrow F_{2g} vibration typical of the Ce-O bond of CeO_2 in the Raman spectra [Fig. 1 (b)]. Underneath, the quick formation of a

α -Al₂O₃ phase together with traces of θ -Al₂O₃ (Cr-fluorescence doublet [41]) is promoted by the Al supply from the γ' -Ni₃Al phase of the γ/γ' substrate. This results in depletion of γ' in the substrate (red line in Fig. 1(a)). The formation of an oxygen defective CeO₂ coating lowers the oxygen partial pressure at the substrate/coating interface and allows the formation of the α -Al₂O₃ as thoroughly explained by Pedraza et al. in [42].

Fig. 1 – (a) Cross-section morphology and (b) overlapped surface Raman analyses of the CeO₂-coated Ni-based superalloy after heat treatment at 1050°C for 1h under flowing Ar atmosphere. NB: The overlapped spectra are taken in different areas for reproducibility purposes.

The surface Raman analyses of the interfacial oxide scale performed in the open cracks [Fig. 1 (b)] also suggest the presence of NiAl₂O₄ spinel phase on top of the alumina scale with its characteristic vibration bands between 600 cm⁻¹ and 1100 cm⁻¹. However, the CeAlO₃

perovskite that Shyu et al. determined through a large fluorescence band underneath the Ce-O vibration [43] is not observed in the areas with the open cracks [Fig. 1 (b)]. This would support our previous hypothesis in which we proposed that the solid-state reaction should only originate at the alumina/ceria interface [44].

3.1- Coated and oxidized conditions

The SEM cross-sections of ceria-coated samples showing the evolution of the oxide scale morphology after isothermal and cyclic oxidation at 1100°C up to 1000h are presented in Fig. 2. Underneath the ceria coating, a duplex oxide scale quite similar to the one observed in Pt-poor γ/γ' or EQ coatings [45,46] is evidenced. The scale consists in a top NiAl_2O_4 spinel phase, which grew mainly by nickel outward diffusion during the early stages of oxidation and before the establishment of a continuous and protective bottom $\alpha\text{-Al}_2\text{O}_3$ scale, as already demonstrated by Pedraza et al. using in-situ XRD diffraction [47]. With exposure time, the alumina subscale grows at the expense of the NiAl_2O_4 but the latter also detaches under cyclic conditions [28]. Once alumina gets in contact with ceria from the coating, an oxide interlayer presenting a nano-grained morphology grows (detailed view in Fig. 2f). In one of our previous studies [28], we considered that this nanograined oxide could correspond to a CeAlO_3 perovskite. This compound can be now confirmed by the presence of a large fluorescence band underneath the Ce-O vibration [Fig. 3 (a)] ascribed to CeAlO_3 (perovskite type) by Shyu et al. [43]. In addition, the main signal of the oxide scale comes from the single $\alpha\text{-Al}_2\text{O}_3$ scale [Fig. 3(b)]. The small Raman shifts of the spinel fingerprints can be related to modifications of its lattice structure and composition.

Complementary tests were therefore performed by oxidizing mixtures of Al_2O_3 and CeO_2 powders at 1100°C for 1h, 3h and 10h under atmospheric pressure of air. The X-ray diffraction patterns of pure CeO_2 , Al_2O_3 and the mixtures $\text{CeO}_2 + \text{Al}_2\text{O}_3$ are shown overlapped in Fig. 4 for comparative purposes. It can be readily observed that no additional peaks related to the perovskite phases appear.

ISOTHERMAL

CYCLIC

Fig. 2 – Cross-sections morphology after oxidation of CeO₂-coated René N5 Ni-based superalloy at 1100°C up to 1000h under isothermal (a,c,e) and cyclic conditions (b,d,f). (a) and (b): 500 h of oxidation. (c) and (d) 1000 h of oxidation. (e) and (f) are respectively, magnified views of (c) and (d).

Fig. 3 – Raman cross-section analyses in the ceria-coated Ni-based superalloy substrate after 1000 h of oxidation at 1100°C in air under isothermal conditions (a) Raman domain, (b) fluorescence domain.

However, the thermodynamic calculations gathered in Figure 5(a) suggest the spontaneity of formation of CeAlO_3 ($\Delta_r G^\circ < 0$) according to equation (1).

One shall note that the reaction can only occur with additional input of Al from the underlying superalloy substrate to CeO_2 and Al_2O_3 . Therefore, DSC measurements were made with $\text{Al}_2\text{O}_3/\text{CeO}_2$ and $\text{Al}_2\text{O}_3/\text{CeO}_2/\text{Al}$ powder mixtures up to 1500°C for two successive heating

(annotated (1) and (2), respectively) and cooling (not presented for the sake of clarity) ramps (Figure 5b). The main thermal events from these DSC curves are gathered in Table 2.

Fig. 4 – X-ray patterns of the pure CeO_2 , Al_2O_3 and the mixtures $\text{CeO}_2 + \text{Al}_2\text{O}_3$ after oxidation at 1100°C in air for 1, 3 and 10 h. Note that no other peaks than those of CeO_2 and of Al_2O_3 appear.

In the absence of Al and for the first heating, the unstable $\gamma\text{-Al}_2\text{O}_3$ of the original powders is observed to transform into the stable $\alpha\text{-Al}_2\text{O}_3$ at a temperature close to the ones that Eisenreich et al. found for the transformations of both γ - and $\theta\text{-Al}_2\text{O}_3$ grown in Al microparticles by high temperature XRD analysis [48]. Thereafter, the relatively weak exothermal event at 1170°C can be associated with the onset of the CeAlO_3 decomposition according to reaction (2) [35]. This implies that a minor amount of CeAlO_3 may have formed under the Ar atmosphere. This reaction also implies that sufficient oxygen is available, which can arise from the oxygen trapped in powders when mixed and put in the crucible. Upon the second heating, only one intense endothermic peak can be observed at 820°C that seem to shadow any other transformation. This peak can be attributed to the reduction of CeO_2 as it coincides with the temperature value retrieved in the temperature-programmed reduction profiles of Piras and coll. [32].

Fig. 5 – (a) HSC Thermodynamic calculation of free energy and heat release during the formation of CeAlO_3 (eq. 1) and (b) DSC measurements of $\text{Al}_2\text{O}_3/\text{CeO}_2$ and $\text{Al}_2\text{O}_3/\text{CeO}_2/\text{Al}$ powders mixtures heated up to 1500°C twice successively.

However, the addition of Al to the CeO_2 and Al_2O_3 brings about many more thermal events, in particular upon the first heating. The melting temperature (658°C) of Al lower than its theoretical one (660°C) can result from the presence of impurities [49]. Like in the absence of Al, the endotherm at 890°C is attributed to the transformation of $\gamma\text{-Al}_2\text{O}_3$ into $\alpha\text{-Al}_2\text{O}_3$ that is fully stabilized at about 1026°C [48]. The increase of temperature results again in the onset of the decomposition of CeAlO_3 . However, the thermal exchange (amplitude of the peak) is far less marked than in the absence of Al. This can be due to the shadowing of the peak at 1026°C

but also to a greater stabilization of the CeAlO₃ in the presence of Al. The second hypothesis can be confirmed upon the second heating with a strong endotherm at 965°C related to the formation of CeAlO₃ that Piras and collab. observed at 950°C upon the reaction of 15% CeO₂ with Al₂O₃ [32], i.e. less CeO₂ than in our study. Further, the melting temperature of Al decreases further (654°C) probably by concentration of the impurities after cooling.

Table 2.- Thermal data for the corresponding DSC curves plotted in Figure 4b.

System	T _{max} ± 2 (°C)	Type	Transformation
Al₂O₃ + CeO₂ (1)	890	endo	γ-Al ₂ O ₃ into α-Al ₂ O ₃ [48]
	1170	exo	Onset of CeAlO ₃ decomposition [35]
Al₂O₃ + CeO₂ (2)	820	endo	Reduction of CeO ₂ [32]
Al₂O₃ + CeO₂ + Al (1)	658	endo	Melting of Al [49]
	890	endo	γ-Al ₂ O ₃ into α-Al ₂ O ₃ [48]
	1026	exo	Full stabilization of α-Al ₂ O ₃ [48]
	1160	exo	Onset of CeAlO ₃ decomposition [35]
Al₂O₃ + CeO₂ + Al (2)	654	endo	Melting of unmolten Al [49]
	965	exo	Formation of CeAlO ₃ [32]

CONCLUSIONS

The oxidation of a CeO₂-coated nickel-based superalloy at 1100°C resulted in the growth of an interfacial CeAlO₃ perovskite phase between the upper CeO₂ and the lower Al₂O₃. The DSC carried out with the mixed powders of CeO₂ and Al₂O₃ (and Al) in Ar (i.e. low oxygen partial pressure) demonstrates that the interfacial pressure underneath the coating shall be also sufficiently low. The DSC results are in line with the thermodynamic calculations and with the observations by microscopy and the Raman analyses. The growth of CeAlO₃ is facilitated with additional Al supply from the substrate. This requires extended exposure at temperature and reduced oxygen partial pressure at the CeO₂ coating/ Al₂O₃ oxide interface and the concomitant disappearance of NiAl₂O₄. The resulting perovskite is made of nanograins. Further transmission electron microscopy studies could be thus performed consolidate our findings.

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