

Promoting exclusive α -Al₂O₃ growth upon high-temperature oxidation of NiCrAl alloys: experiment versus model predictions

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Abstract

The relationships between the compositional changes in the alloy and the morphology and phase constitution of the developing oxide scale are established as a function of oxidation time for the thermal oxidation of a γ -Ni–27Cr–9Al (at.%) alloy at 1373 K and a high and low partial oxygen pressure (pO_2) of 2×10^4 and 0.1 Pa, respectively. Based on experimental observations, and model calculations using a coupled thermodynamic-kinetic oxidation model, four successive growth stages can be identified for the oxidation of a γ -Ni–27Cr–9Al alloy at 1373 K. It is shown that the exclusive growth of α -Al₂O₃ is promoted for oxidation at low pO_2 by maintaining a sufficiently high concentration of Al in the alloy at the oxide/metal interface during the initial stages of fast oxide-scale growth.

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1. Introduction

In state-of-the-art thermal barrier coating (TBC) systems for gas turbine applications, MCrAlY (M = Ni, Co) alloys are used as bond coatings between the thermal insulating ceramic topcoat and the underlying, load-bearing superalloy component. The application of a bond coat is required to protect the component against oxidation and corrosion at high temperatures (>1300 K) and to provide the necessary adhesion of the component with the ceramic topcoat [1]. MCrAlY coatings generally have a dual phase structure, consisting of an Al-rich β -phase (body-centered cubic) and an Al poor γ -phase (face-centered cubic) [1]. The protection offered by such

an alloy against high-temperature oxidation, relies on the ability of the alloy to produce and maintain a stable, continuous, slow growing and adherent oxide scale on its surface. These requirements are met best by a continuous α -Al₂O₃ scale, because the effective diffusion rates of oxygen and metal ions (e.g. Al, Cr, Ni) through α -Al₂O₃ are relatively low and the chemical and thermal stability of α -Al₂O₃ is relatively high [2].

However, at the onset of high-temperature oxidation of as deposited MCrAlY coatings, the exclusive formation and growth of such a protective α -Al₂O₃ scale normally does not occur. The initial stages of oxidation of MCrAlY alloys generally proceed by the simultaneous formation of α -Al₂O₃ and fast growing, non-protective oxide phases such as Cr₂O₃, NiO, NiCr₂O₄ and NiAl₂O₄ [3–7]. These non-protective oxide phases promote the spalling of the ceramic topcoat from the bond coat upon thermal cycling, which results in rapid degradation of the underlying superalloy component [8,9]. Therefore, ideally, the formation of an oxide scale

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solely constituted of α -Al₂O₃ is preferred on the bond coat surface.

To control the exclusive growth of α -Al₂O₃ on a MCrAlY alloy at the onset of oxidation requires fundamental knowledge on the relationship(s) between the oxide growth mechanism(s) and the corresponding composition and microstructure of the developing oxide scale and the underlying alloy. To date, most studies on the high-temperature oxidation of MCrAlY alloys have focussed on the effect of the bulk alloy composition on the sequence of formation and the resulting distribution of the different oxide phases within the oxide scale [3–7,10,11]. Only recently, a coupled thermodynamic-kinetic (CTK) oxidation model has provided detailed knowledge on the delicate interplay between the oxide-scale growth kinetics, the development of oxide phases and the compositional changes induced within the underlying alloy [12]. The results of these model calculations show that the formation of undesired, non-protective oxide phases is governed by the compositional changes within the underlying alloy, in particular the concentrations of the alloy constituents in the alloy at the oxide/metal (O/M) interface. It followed that the exclusive growth of α -Al₂O₃ on a MCrAlY alloy can be promoted by maintaining a sufficiently high concentration of Al in the alloy at the O/M interface during the initial stages of very fast oxide-scale growth [12].

In this study, the relationships between the compositional changes in the alloy and the morphology and phase constitution of the developing oxide scale were established as function of oxidation time for the oxidation of a γ -Ni–27Cr–9Al (at.%) alloy at 1373 K and a high and low partial oxygen pressure (p O₂), respectively. To this end, the γ -Ni–27Cr–9Al alloy was oxidised between 5 min and 64 h at a p O₂ of 2×10^4 and 0.1 Pa in a furnace and an UHV processing chamber, respectively. The structure, morphology and thickness of the grown oxide scales, as well as the amount of each oxide phase developed as function of oxidation time, were investigated by scanning electron microscopy (SEM). The phase constitution and composition of the developing oxide scale were determined by X-ray diffractometry (XRD), energy-dispersive X-ray spectrometry (EDXS) and Auger electron spectroscopy (AES). Utilising the high spatial resolution in scanning Auger microscopy (SAM), the concentrations of Al and Cr in the alloy at the O/M interface were determined as function of oxidation time. The experimentally obtained values for the interface concentrations in the alloy and the amount of each oxide phase developed as function of oxidation time were compared with their corresponding calculated values, as obtained from the recently developed CTK oxidation model [12]. The role of the p O₂ as a tool to promote the exclusive formation of α -Al₂O₃ is discussed.

2. Experimental

2.1. Material and specimen preparation

High purity (>4 N) Ni, Cr and Al were used in the preparation of a single-phase γ -Ni–27Cr–9Al (at.%) alloy. The alloy was cast in the form of a cylindrical rod and subsequently annealed in a sealed quartz tube filled with Ar at 1373 K for 400 h in order to homogenize and recrystallize the alloy. The average composition after annealing, determined using quantitative [13] electron probe X-ray microanalysis (EPMA), employing wavelength-dispersive spectrometry (WDS), was Ni–27.1Cr–8.9Al (at.%).

Next, disc-shaped specimens (diameter 10 mm and thickness 2 mm) were cut using spark-erosion. Prior to oxidation, the specimen surface was prepared by successively grinding and polishing. Polishing was performed with paste of 3, 1 and 0.25 μ m diamond grains. Hereafter, the surface was fine-polished, using a chemical resistant cloth with a suspension of 0.04 μ m silica in distilled water. After each preparation step the specimens were thoroughly cleaned ultrasonically with isopropanol and dried by blowing with pure compressed nitrogen gas.

After oxidation and subsequent XRD analysis (see Section 2.3), oxide/alloy cross-sections were prepared. To protect the oxide scale from spalling during preparation, first a copper layer was deposited on the grown oxide scale by evaporation. Then, a nickel layer was plated on top of the copper layer using a Watts bath. Next, the specimen was cut perpendicular to the specimen surface with a diamond saw. Finally, the oxide/alloy cross-sections were ground and polished as described above.

2.2. Oxidation

Oxidation experiments at a high p O₂ of 2×10^4 Pa were performed in a horizontal alumina tube furnace (Lenton PTF 16/75/610) at 1373 K. A gas mixture of argon (99.998 vol% Ar) and 20 vol% oxygen gas (99.998 vol% O₂) was passed through the furnace tube with an inner diameter of 75 mm, at a total pressure of 10^5 Pa, with a controlled flow rate of 750 ml/min. With the furnace at its oxidising temperature of 1373 K, the specimens were placed in an annealed alumina crucible and transported within several seconds to the hot zone of the furnace. Thus, the onset of oxidation corresponds with the time at which the specimen is introduced into the furnace. Exposure times varied between 10 min and 64 h. After oxidation, the specimens were transported quickly out of the hot zone of the furnace and allowed to cool for 5 min in ambient air.

Oxidation experiments at a low p O₂ of 0.1 Pa were performed in an UHV processing chamber (base pressure $<10^{-7}$ Pa). Prior to oxidation, the native oxide film

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