

Silicon based oxidation-resistant coatings on Ti6242 alloy by dynamic ion mixing

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Abstract

The influence of Si_xC_y and Si_xN_y amorphous coatings on the oxidation resistance of a Ti6242 (Ti–6Al–2Sn–4Zr–2Mo) alloy was investigated. They were produced at room temperature by the dynamic ion mixing technique combining physical vapour deposition with simultaneous bombardment with 120 keV Ar^+ ions. Isothermal oxidation tests were carried out at 600 °C in 1 atm flowing synthetic air (80% N_2 , 20% O_2) demonstrating a considerable reduction (~two orders of magnitude) of the oxidation rate for at least 100 h. The structural modifications after oxidation were investigated by XPS, XRD, SEM, SIMS. The formation of SiO_2 is detected as the main oxidation product in the coating and the formation of Ti–Si compounds is also observed in the coating/substrate interface region. The crystallisation of Si_xN_y is not detected and for Si_xC_y only some traces of β -SiC could exist. The improvement of oxidation resistance of Ti6242 is discussed in relation with the intrinsic properties of the coatings and with the interface mixing and ion beam densification.

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1. Introduction

The high strength and resistance to both fatigue and creep make modern titanium alloys attractive materials for the compressor blades of gas turbine aero engines operating at temperatures above 500 °C. However at these temperatures the titanium alloys and particularly those of α/β mixed structure are susceptible of rapid oxidation in air as well as α -case formation [1–3] because the migration of oxygen promotes the formation of an oxygen-rich Ti hexagonal solid solution (α -phase) and modifies the α/β mixed phase structure near the surface. These modifications due to the inward diffusion of oxygen and particularly the preferential stabilization of the more brittle alpha-phase considerably reduce the mechanical properties of titanium

alloys above 500 °C by the increased tendency of crack formation under stress. The research for improving the oxidation resistance of titanium based alloys by various surface treatments and protective coatings has been very active and is stimulated by the need to improve both the durability and engine performance in aerospace industry [4].

A good reduction in oxidation of pure Ti has been obtained by a thin Si coating bombarded with Ar^+ ions to produce interface mixing with the substrate [5], siliconizing at high temperature appears also a promising technique for improving the oxidation resistance of γ -TiAl [6,7], silicon nitride ceramic coatings have been also previously applied successfully for improving oxidation resistance of titanium alloys [8,9]. It appears that silicon based ceramic coatings exhibit interesting physicochemical properties for oxidation protection of titanium alloys. However they must fulfil two important requirements: strong adhesion and high density (i.e. low porosity or cracks). This means that the durability

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and performance of a coating will depend critically not only on its intrinsic properties but also on the deposition technique.

It has been demonstrated that the bombardment of a growing film with either low energy Ar^+ ions (6 keV) [10,11], or high energy Ar^+ ions (60–300 keV) [12,13], produces beneficial modifications in the film structure and interface with the substrate. The process of Ion Beam Assisted Deposition (IBAD) involves bombardment with a low energy ion beam (3–15 keV) simultaneously with the deposition and enables the formation of dense coatings. The Dynamic Ion Mixing (DIM) is a variant of IBAD using ion beams of higher energy (100–300 keV) to achieve a more efficient interface mixing. The spatial redistribution and atomic transport at the coating/substrate interface is produced at room temperature over relatively long distances and creates an interface of graded composition improving the adhesion of the coating [13,14]. The high energy ion bombardment can also modify the structure of the growing film as a result of the high density of displacements and replacements of atoms in collision cascades. These very specific effects contribute to the formation of dense and adherent ceramic coatings on metallic surfaces which are of special interest for corrosion protection applications.

In the present study we have investigated the influence of SiC and Si_xN_y coatings deposited by DIM on the oxidation resistance of a Ti6242 alloy at 600 °C.

2. Experimental conditions

2.1. Material and surface preparation

The Ti6242 alloy exhibits the α/β mixed phase structure analogous to the more conventional TA6V alloy but it can be considered as a nearly α -alloy. The chemical composition is Ti–6Al–2Sn–4Zr–2Mo, the numbers are in weight percentage and the balance is titanium. The samples used for the oxidation experiments consist in discs 15 mm in diameter and 2 mm in thickness. In order to investigate the influence of the surface roughness we have prepared two series of samples: in a first one (sample A) the samples were mechanically polished up to grit 500 on both sides as well as the lateral surface to a mean roughness $R_a \sim 0.1 \mu\text{m}$, in a second series (sample B) mechanical polishing was performed up to grit 4000. Then the surfaces were polished up to a “mirror” finish using a 1 μm diamond paste, giving a final roughness $R_a \sim 0.03 \mu\text{m}$. All the samples were ultrasonically cleaned in acetone and ethanol and finally dried.

2.2. Coating deposition conditions

Si_xC_y and Si_xN_y coatings were deposited at room temperature (RT) by ion beam sputtering using a Kaufman type ion source of 7.5 cm diameter and the samples are

mounted on a rotating substrate holder. A water cooled target consisting of a sintered disk either SiC or Si_3N_4 of 10 cm diameter was sputtered under a 45° angle of incidence with 1.2 keV Ar^+ ions. The pressure of the chamber before the deposition was $5 \cdot 10^{-5}$ Pa and was maintained at this pressure during the deposition procedure. All the external surfaces of the samples, including the lateral ones were uniformly coated. The procedure consisted in depositing firstly the Si_xC_y or Si_xN_y coating on the front disc surface; then the chamber was opened and the samples were turned up to deposit on the second disc side; since there is no shadowing of the lateral disc surfaces they are also coated. A calibrated quartz crystal oscillator was used to monitor the growth rate and the final film thickness. The ion beam mixing was performed with 120 keV Ar^+ ions during all the film deposition for Si_xC_y coatings and only on the first 40 nm of deposit for those of Si_xN_y . The measured final film thicknesses were respectively 0.21 μm for Si_xN_y and 0.25 μm for SiC.

2.3. Oxidation tests and characterization methods

The chemical composition of Si_xC_y and Si_xN_y coatings were determined by Rutherford Backscattering Spectrometry and also by XPS. The modification of the surface composition of the coatings was investigated using X-ray photoelectron spectrometer (XPS, ESCALAB MK II). The samples were introduced in the apparatus low pressure chamber (10^{-8} Pa) 12 h before analysis and we used an Ar focussed beam for cleaning the surface of the samples in order to remove surface contamination due to laboratory air. Experiments were carried out using the Mg K_α X-ray radiation ($h\nu = 1253.6$ eV) and the following binding energies were selected: Al–2p, Si–2s; C–1s; Ti–2p; O–1s.

Depth profile distributions were also performed by glow discharge optical emission spectroscopy (GDOES) and by SIMS experiments (CAMECA IMS4F6 apparatus using Cs^+ ions). Secondary Ion Mass Spectroscopy (SIMS) was used to characterize the coated samples before and after oxidation; surface sputtering was realized with 10 keV Cs^+ ions and a 30 nA current intensity. Cesium atoms, were chosen in order to produce negative ions from electro-negative elements such as $\text{O}^{2-}\text{Zr}^{2-}$.

High temperature oxidation tests have been conducted in SETARAM TAG24S equipment at 600 °C. This apparatus has the advantage of combining good accuracy with a limitation of buoyancy effects due to a symmetrical furnace in which an inert sample counterbalances these effects. Isothermal oxidation tests have been carried out in 1 atm flowing synthetic air (80% N_2 , 20% O_2). Heating and cooling rates were always of 1°/s. Dwell time for all the tests was 100 h. The weight gain was continuously measured as a function of time. In this study, oxidation kinetics during isothermal tests has been analyzed following the procedure detailed in [15].

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