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Original Research Article

Microstructure and positron lifetimes of zirconium modified aluminide coatings

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ABSTRACT

The microstructure of the zirconium modified and non-modified aluminide coatings was examined by the EDS, XRD, TEM and the positron annihilation spectroscopy methods. Both coatings have a double layer structure: β -NiAl phase on the top and γ -Ni₃Al below. Small zirconium nanoparticles were found along grain boundaries in the β -NiAl phase. The positron lifetime in both coatings is the same. The formation of zirconium precipitates neither affects, the number of defects nor the volume diffusion. Zirconium nanoparticles that precipitate along grain boundaries stand against the outward diffusion of Al ions through the coating to the coating/oxygen interface. This “blocking effect” may be responsible for the reduction of the alumina scale growth rate and may delay pore formation on the coating/oxygen interface.

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1. Introduction

Protective coatings have a wide range of applications. For instance they are used as protective layers of the hot section of aircraft engine made of nickel superalloys. In this case protective aluminide coatings are applied in order to increase oxidation resistance of treated parts [1]. The oxidation resistance of the coating and its adherence to the substrate can be improved by the slow oxide scale growth [2]. It could be achieved by the incorporation of noble metals, particularly platinum [3]. Platinum acts as NiAl phase stabilizer and prevents the β to γ phase transformation [4]. Moreover, it

encourages formation of pure alumina oxide and reduces oxide growth rate [5]. The increase of oxidation resistance could also be achieved by the addition of small amount of reactive elements (RE) such as: hafnium [6], zirconium [7], yttrium and cerium [8,9]. According to Ning et al. [10] NiAl-based coatings containing small quantities of reactive elements (Hf or Zr) added either in conjunction with or in place of Pt exhibit better oxidation resistance than nonmodified ones.

According to Li et al. [11] small Zr addition (about 0.1 at%) improves coatings' oxidation resistance due to the diminishing of the scale spalling. However, General Electric Company developed several new coatings containing much higher R.E.

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additions, i.e. 2 at% hafnium and/or zirconium and up to 5 at% chromium [6,12]. Zirconium, homogeneously distributed within the oxide scale after cyclic oxidation, delays spallation and improves the scale adhesion [13]. Other elements (such as Sm, Gd, Yb, Sc [14] Dy [15]) cannot only reduce the oxidation rate of NiAl coatings but also enhance the oxide scale adhesion to the substrate. Hafnium and yttrium added to the substrate improved oxidation resistance of the coated alloy and scale adhesion [16]. Research performed by the Li et al. [11] revealed that RE ions first segregate to the scale/alloy interface and then diffuse to the oxide scale surface along oxide grain boundaries. The slow outward diffusion of large RE ions stands against the outward diffusion of aluminum ions and inward diffusion of oxygen. Accordingly, it is called "the blocking effect" and it is supposed to be responsible for the reduction the alumina scale growth rate [17]. Nevertheless some aspects of this mechanism remain problematic. For instance, it is suggested that zirconium (less than 1 at.%) could contribute to the faster reduction of Al vacancies [18] through fast diffusion of Al toward the metal/oxide interface [19].

Positron annihilation spectroscopy is a well-established tool for the microstructure investigation of condensed matter. Positrons implanted into a metallic system can be localized in regions where electron density is lower than in a perfect crystal lattice, i.e. open volume defects, and then annihilate with electrons [20]. In the case of vacancies and small vacancy clusters the relationship between the positron lifetime and the size of open volume defects is well-known [21]. In the case of alloys, the annihilation of positrons with the localized electrons of atoms surrounding open volume defects deliver unique signature which may be used for their identification [22]. That is why, positron lifetime spectroscopy can also shed light on the interactions between open volume defects and alloying components' atoms, which is important for structural materials containing many elements.

In the research presented in this paper, the positron spectroscopy has been applied for the first time to analyze the zirconium influence on the structure of defects in aluminide coatings. Moreover, the microstructure analysis was performed by the SEM, XRD and TEM techniques.

2. Experimental

Circular specimens of pure nickel (99.95 wt.%) 20 mm diameter and 4 mm high were used as the substrate. Specimens were grounded up to SiC No 1000, degreased in ethanol, ultrasonically cleaned and finally aluminized. Coatings were deposited using the CVD equipment BPXPR0325S manufactured by the Ion Bond company (Fig. 1) [5,23].

The non-modified coating was deposited for 360 min at 1020 °C, whereas the zirconium-modified coating was deposited in the several stages such as: heating from the room temperature up to 1020 °C, aluminizing at 1050 for 30 min, aluminizing and zirconizing for 90 min, then aluminizing for 240 min. Aluminum chloride vapor (AlCl_3) was produced in an external generator and transported into the CVD reactor, where samples were placed. AlCl_3 vapor reacted with nickel, and grains of intermetallic phase (NiAl) were formed according to the reaction:

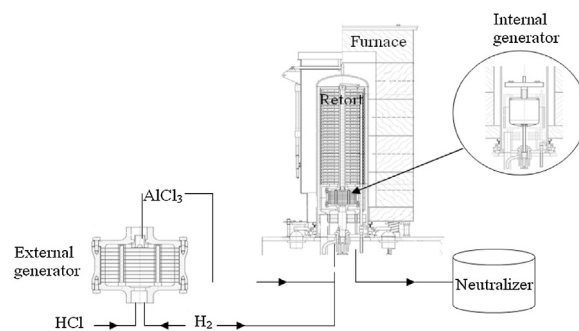
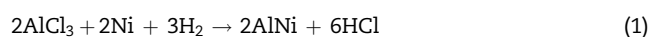


Fig. 1 – A scheme of equipment for deposition of aluminide coatings by the CVD method [23].



When the zirconium-modified coating was deposited, zirconium chloride vapor ZrCl_3 was produced in an external generator and transported into the CVD reactor, where the nickel sample was placed. AlCl_3 and ZrCl_3 vapors reacted with nickel at 1020 °C. The precise description of this process is presented in Ref. [23].

The microstructure of the cross-sections of two coatings was examined by an optical microscope Nikon Epiphot 300, a scanning electron microscope (SEM) Hitachi S-3400N and an energy dispersive spectroscope (EDS). The coating thickness was determined by means of the NIS-Elements software. Phase composition of the coating was investigated by the use of the ARL X'TRA X-ray diffractometer, equipped with a filtered copper lamp with the voltage of 45 kV. A Tecnai G2 STWIN FEG (200 kV) transmission-scanning electron microscope equipped with the High Angle Annular Dark Field Detector (HAADF) was used for the microstructure investigation. Thin foils were cut perpendicularly to the surface with the Quanta Dual Beam Focused Ion Beam (FIB) equipped with the Omniprobe lift-out system.

Positron lifetime measurements were performed for initially annealed nickel at 1200 K and coated samples. The positron lifetime spectra of more than $1.5 \cdot 10^6$ counts were measured using the fast-fast spectrometer with BaF_2 scintillators. The time resolution of the system was 265 ps (FWHM). The 70-mCi activity positron source containing ^{22}Na isotope enveloped in 7- μm thick kapton foil was sandwiched between samples. The average implantation depth (which is the reciprocal value of the linear absorption coefficient) for positron emitted from ^{22}Na into aluminum and nickel is about 87 and 24 μm , respectively. All obtained spectra were analyzed using the LT code [24] with background and source corrections.

3. Results and discussion

The SEM image of zirconium doped aluminide coating deposited for 240 min by the CVD method at 1020 °C is presented in Fig. 2.

The coating has a double layer structure. In the first layer located on the top of the coating, the proportion of Ni to Al corresponds to the β NiAl phase (Table 1, point 1).

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