



Effect of $\text{La}_2\text{O}_3/\text{CeO}_2$ particle size on high-temperature oxidation resistance of electrodeposited $\text{Ni-La}_2\text{O}_3/\text{CeO}_2$ composites

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Abstract: $\text{Ni-La}_2\text{O}_3/\text{CeO}_2$ composite films were prepared by electrodeposition from a nickel sulfate bath containing certain content of micrometer and nanometer $\text{La}_2\text{O}_3/\text{CeO}_2$ particles. The effect of La_2O_3 or CeO_2 particle size on the oxidation resistance of the electrodeposited $\text{Ni-La}_2\text{O}_3/\text{CeO}_2$ composites in air at 1000 °C was studied. The results indicate that, compared with the electrodeposited Ni-film, $\text{Ni-La}_2\text{O}_3/\text{CeO}_2$ composites exhibit a superior oxidation resistance due to the codeposited La_2O_3 or CeO_2 particles blocking the outward diffusion of nickel. Moreover, compared with nanoparticles, La_2O_3 or CeO_2 microparticles have stronger effect because La_2O_3 or CeO_2 microparticles also act as a diffusion barrier layer at the onset of oxidation.

Key words: Ni-based composite; electrodeposition; high-temperature oxidation; La_2O_3 ; CeO_2 ; reactive element effect; oxidation mechanism

1 Introduction

Electrodeposition is a low-cost and low-temperature method suitable for producing metal matrix composite materials [1–3], which have potential applications in the aerospace industry due to their high temperature oxidation resistance [4,5]. In this process, fine particles or whiskers are suspended in the electrolyte and embedded in the growing metal layer. Recently, a considerable amount of research effort has been put into electrodeposition of Ni-based composite. With the codeposition of small amount of reactive elements oxides, such as Y_2O_3 , CeO_2 and La_2O_3 , the oxidation resistance of Ni film can be further improved [4–9] because the rapid outward diffusion of Ni along the grain boundary was inhibited mainly by segregated RE ions. The phenomenon was first reported in 1937 [10] and was referred to as “reactive element effect (REE)”. Various theories to elucidate the REE have been put forward but still are in dispute because the mechanism may differ for different oxide/RE systems [11,12]. PENG et al [4,5] further confirmed that only when La_2O_3 particles are

small enough, they may act as a source of La ions by solution at high temperature to block the outward diffusion of Ni. The results were supported by works of CZERWINSKI et al [13,14], who proved that the addition of a sol-gel coating of ceria particles below 10 nm onto a nickel surface retarded the oxidation; however, when the size of CeO_2 particles increased over some size limit by sintering and coarsening, the oxidation of nickel actually increased. Recently, ZHOU et al [9] have found that the codeposited Y_2O_3 microparticles could significantly improve the oxidation resistance of Ni film because the re-precipitation of some Y_2O_3 nanoparticles along the grain boundaries blocked the outward diffusion of Ni and changed its oxidation mechanism. It seems that the codeposited RE oxide microparticles should have the same effect as the nanoparticles counterpart. In view of current production cost and commercial availability of RE oxide nanoparticles, the application of microparticles rather than nanoparticles to electroplate oxidation-resistant Ni-film is of great interest from the point of engineering view. Therefore, it is useful to know the effect of RE oxide particle size on the oxidation of Ni films.

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However, there has been no report about these yet. Previous works [4–7] also indicated that, in order to improve the oxidation resistance, the addition of a certain content of RE oxide nanoparticles is necessary. Lower content can not supply enough RE ions in short time to block the outward diffusion of Ni, while higher content even accelerates the oxidation of Ni film. XU et al [7] found 2.7% CeO₂ (mass fraction) nanoparticles could improve the oxidation of Ni-film. PENG et al [4,5] reported that 8.2% La₂O₃ (mass fraction) particles could also improve the oxidation of Ni-film. In this work, nickel composites with (6.5±0.5)% La₂O₃ or (4.5±0.4)% CeO₂ were chosen for the oxidation experiment to investigate the effect of RE oxide (La₂O₃ or CeO₂) particle size on the oxidation resistance of electrodeposited Ni–La₂O₃/CeO₂ composites.

2 Experimental

Pure nickel specimens with dimensions of 15 mm×10 mm×2mm were cut from a pure electrolytic nickel plate and then were abraded with 800# grit SiC waterproof paper. After being ultrasonically cleaned in acetone, the specimens were electrodeposited with (on all sides) a 20–55 μm-thick film of Ni–La₂O₃ or Ni–CeO₂ composite from a nickel sulfate bath containing 150 g/L NiSO₄·7 H₂O, 15 g/L NH₄Cl, 15 g/L H₃BO₃, 0.1 g/L C₁₂H₂₅NaSO₄, and 25 g/L pure La₂O₃ or CeO₂ particles, respectively. The mean diameters of micrometer La₂O₃ and CeO₂ particles were 1.5 and 2.5 μm, respectively. The mean diameters of nanometer La₂O₃ and CeO₂ particles are 80 and 10 nm, respectively. Electrodeposition was conducted in 1000 mL beaker at a current density of 3 A/dm², temperature of 35 °C and pH value of 5.5–6.0. During the electrodeposition, magnetic stirring was employed to maintain the uniform particles concentration and prevent the sedimentation. The detailed coating process could be found elsewhere [4,5]. For comparison, a 55 μm-thick Ni film was also deposited under the same parameters in the same bath but without adding any La₂O₃ or CeO₂ particles.

The isothermal oxidation experiments were carried out in air at 1000 °C for 20 h. The mass measurements were conducted after fixed time intervals using a balance with a sensitivity of 0.01 mg. The composition and phases of the as-deposited films before and after oxidation were investigated using a Camscan MX2600FE type scanning electron microscope (SEM) with energy dispersive X-ray spectroscopy (EDS) and D/Max–2500 pc type X-ray diffraction (XRD). The average nominal La₂O₃ or CeO₂ concentrations were determined by using the La or Ce to oxygen ratio according to its chemical formula at 1000 magnification. Ten replicate tests at different locations were carried out

so as to minimize data scattering, and every value reported was an average of ten measurements. Electroless Ni-plating was plated on the surface of the oxidized specimens to prevent the spallation of the scales for observing cross-sections.

3 Results

A regular pyramidal structure as shown in Fig. 1(a) is observed on the surface of the Ni film due to a typical Ni growth texture [15]. However, with the addition of La₂O₃ or CeO₂ particles, the grain size is significantly reduced and the morphology is changed to hemispherical grain structure except the one with CeO₂ microparticles, which have a nodular grain structure, as seen in Figs. 1(b) and (d). EDS results show that both the as-deposited Ni–La₂O₃ composites contain (6.5±0.5)% La₂O₃ (mass fraction) and the as-deposited Ni–CeO₂ composites contain (4.5±0.4)% CeO₂ (mass fraction). Although the distribution of bright La₂O₃ or CeO₂ microparticles can be observed in some locations, the particles are not present in most areas, the same as the previous reports [4,5]. At the same time, lots of La₂O₃ microparticles and CeO₂ nanoparticles form agglomerate clusters, as shown in Figs. 1(c) and (e). Figure 2 shows the cross sectional morphologies of the as-deposited Ni–La₂O₃ and Ni–CeO₂ nanocomposites with a thickness of about 20 μm. The EDS results show that the white spots are enriched in La or Ce, implying that the deposited La₂O₃ or CeO₂ nanoparticles are partially agglomerated.

The oxidation kinetics of various samples at 1000 °C for 20 h is illustrated in Fig. 3 as linear plots (Fig. 3(a)) and parabolic plots (Fig. 3(b)), respectively. At 1000 °C, all samples obey the parabolic rate law to a good approximation for the whole duration of the test. The calculated parabolic rate constants are listed in Table 1. It is obvious that the addition of La₂O₃ or CeO₂ particles improves the oxidation resistance of Ni film. However, La₂O₃ or CeO₂ microparticles have stronger effect to decrease the oxidation rate of Ni. From Table 1, it can be found that, compared with La₂O₃ particles, CeO₂ particles have stronger effect.

XRD analysis shows that the scales formed on all samples are NiO. Figure 4 shows the surface morphologies of various samples after oxidation. Faceted NiO grains with the mean grain size of around 10 μm appear on the Ni film, as seen in Fig. 4(a). However, the NiO crystals formed on the Ni–La₂O₃ or CeO₂ composites are fine grains, especially the composites with La₂O₃ or CeO₂ microparticles, as seen in Figs. 4(c) and (d). From Fig. 4(b), it can be found that the scales are also somewhat convoluted. From the corresponding cross-section, the scales formed on Ni film exhibit a double-layer structure with a similar thickness: a

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