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Abstract

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Keywords

Ames Laboratory, Coating, Microstructure, Transmission electron microscopy (TEM)

Disciplines

Materials Science and Engineering | Metallurgy

Comments

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Microstructure investigations of Pt-modified γ'-Ni_3Al + γ-Ni coatings on Ni-based superalloys

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The microstructure of Pt-modified γ'-Ni_3Al + γ-Ni coating on CMSX-4 single-crystal superalloy has been investigated by transmission electron microscopy (TEM). Cross-sectional TEM analyses showed the presence of precipitates in the coating. This precipitate was identified as the hexagonal topologically close-packed (TCP) μ phase with lattice parameters $a = 0.473$ nm and $c = 2.565$ nm. The energy-dispersive x-ray (EDX) spectrum of the μ phase suggested a refractory element rich compound comprising the elements Re, W, and Co. Twin domains parallel to (001) were found in the μ phase. The mechanisms of the μ phase and twinning formation were discussed.

I. INTRODUCTION

The drive for larger power, higher efficiencies, longer lifetimes, and reduced emissions has led to more severe working environments for the components in advanced gas turbine engines. The improved performance of Ni-based superalloys has been achieved by novel materials design, improved cooling technologies, and better manufacturing methods; however, the potential for raising the operating temperature of superalloys still higher is limited. The use of thermal-barrier coatings (TBCs) coupled with internal cooling of the underlying superalloy components is capable of providing temperature reduction of about 100 to 300 °C, while simultaneously increasing oxidation and corrosion resistance. This has enabled modern gas turbine engines to operate with a gas stream temperature higher than the melting temperature of the superalloys, thereby achieving a substantial improvement in efficiency and performance.[1–7]

The state-of-the-art TBCs are three-layered structures, consisting of a ceramic top coat, a thermally grown oxide (TGO), and an oxidation-resistant bond-coat alloy that is applied to a nickel-based superalloy component. Most of the ceramic top coat of TBCs is ZrO_2 stabilized with 7 to 8 wt% Y_2O_3 (YSZ) applied by either air plasma (APS) spraying or electron beam physical vapor deposition (EB-PVD). The bond coat is a diffusion-processed β-(Ni,Pt)Al aluminide (a platinum-modified β-NiAl) or a MCrAlY overlay coating, where M is Ni, Co, or a mixture of Ni and Co.[1–6] The microstructure of MCrAlY mainly consists of β-Ni(Co)Al and γ-Ni(Co,Cr) phase.[8] The columnar microstructure of the YSZ deposited by EB-PVD and the high oxygen permeability of the YSZ require that the bond coat be able to form a protective, stable, and slow-growing thermally grown oxide (TGO) to prevent oxidative attack of the alloy.[1–6] Current commercial state-of-the-art β-(Ni, Pt)Al aluminide or MCrAlY overlay bond coatings form an alumina scale; however, the continued growth of a TGO-Al_2O_3 scale will result in aluminum depletion from the bond coating during high-temperature exposure. As aluminum is depleted, the average composition of the bond coat becomes increasingly enriched in nickel until reaching the single-phase boundary, at which point further depletion leads to the formation of γ'-Ni_3Al. On the other hand, the bond coat transforms from its as-fabricated β-(Ni,Pt)Al (*B*2) structure to a β′ (*L*10) martensite during heating and cooling.[5,6] The volume changes accompanying this transformation stress the bond coat and cause undulation and delamination of ceramic coatings.

Recently Gleeson et al.[9,10] patented Pt-modified γ'-Ni_3Al + γ-Ni coatings showing improved high-temperature oxidation and corrosion resistance compared with β-(Ni,Pt)Al and MCrAlY overlay coatings. The novel Pt-modified γ'-Ni_3Al + γ-Ni bond coats are therefore more compatible with the underlying substrate and provide a metallurgical stable microstructure to avoid phase transformations, such as β-NiAl → γ'-Ni_3Al and β-NiAl (B2) → β′ (*L*10) that have been observed to occur in β-(Ni,Pt)Al and MCrAlY overlay coatings. Simple Pt-modified $\gamma' + \gamma$ coatings were synthesized on superalloys by electroplating a layer of Pt followed by a

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diffusion treatment.[11–18] By introducing a secondary short-term aluminizing step via pack cementation or chemical vapor deposition (CVD), Al-enriched Pt-modified $\gamma' + \gamma$ coatings were achieved.[9,10,19–22]

Most of the investigations into Pt-modified γ'-Ni_3Al + γ-Ni coatings have focused on coating preparation, hot corrosion and oxidation, and interdiffusion behavior.[13–23] Significant quantities of refractory elements (such as rhenium and tungsten) are added to second- and third-generation, single-crystal Ni-based superalloys to reduce the coarsening rate of the strengthening γ'-Ni_3Al phase giving improved high-temperature mechanical properties, but tend to promote microstructural instabilities and formation of topologically close-packed (TCP) phases.[7,24–26] The TCP phases are typically rich in refractory alloying elements and possess complex crystal structures characterized by close-packed layers of atoms. Four different TCP phases, including the tetragonal σ phase ($P4_2/mnm$), rhombohedral R and μ phases ($R\bar{3}m$), and orthorhombic P (Pnm) phases have been observed in Ni-based superalloys.[25,26] Interdiffusion occurs during the coating preparation process, as well as later during high-temperature exposure in service. Both can affect coating microstructure and, in turn, high-temperature performance.[17,27] So far, detailed microstructural investigations on Pt-modified γ'-Ni_3Al + γ-Ni coatings on single-crystal Ni-based superalloys, in particular transmission electron microscopy (TEM) characterization, have been limited.

In the present work, we report on cross-sectional view TEM investigations of a platinum-modified γ'-Ni_3Al + γ-Ni coating on CMSX-4 single-crystal superalloy substrate (Cannon Muskegon Corp., Muskegon, MI). Such a study is expected to be helpful for understanding the growth processes of bond coats and relationships between their microstructure and properties.

II. EXPERIMENTAL

Platinum-modified γ'-Ni_3Al + γ-Ni coatings are manufactured first by electroplating ~7 μm of platinum on CMSX-4 substrate, then heat treating to diffuse the platinum into the nickel-based superalloy prior to aluminizing. The aluminizing process was performed via pack cementation. The packs consist of NH_4Cl activator, a source of aluminum and hafnium, and inert Al_2O_3 filler powder.[9,10]

Cross-sectional TEM specimens were prepared by a standard technique involving cutting and then gluing face to face before mechanical grinding and polishing, dimpling, and ion milling to perforation. TEM bright field imaging, selected-area electron diffraction (SAED), and energy-dispersive x-ray (EDX) analyses of the sample were performed using a Philips CM30 electron microscope equipped with a LaB_6 electron source, operated at an acceleration voltage of 300 kV.

III. RESULTS AND DISCUSSION

A scanning electron micrograph of the as-coated Pt + γ'-Ni_3Al + γ-Ni coating on the CMSX-4 substrate is shown in Fig. 1(a). The coating is about 30 μm thick. Voids[17,22] were seen in the substrate adjacent to the coating. Figure 1(b) shows a typical two-phase (γ'-Ni_3Al + γ-Ni) microstructure for CMSX-4 superalloy substrate. The γ'-Ni_3Al precipitate (~500 nm) has a cuboidal morphology with unimodal distribution in the γ-Ni matrix. The inset is the SAED pattern of one γ' precipitate showing the γ' along the [001] zone axis. The γ' phase has $L1_2$ structure with lattice parameter $a = 0.357$ nm. Electron diffraction showed no MC (M = metal atom) carbides or TCP phases in the superalloy substrate.[7,28–30]

A representative TEM microstructure of the as-coated Pt + γ'-Ni_3Al + γ-Ni coating is shown in Fig. 1(c). Figure 1(d) is the SAED pattern of region A in Fig. 1(c), corresponding to the $[1\bar{1}0]_{\gamma'}$ zone axis. The electron-diffraction patterns of regions B and C reveal that both regions B and C are γ'-Ni_3Al. Precipitates having characteristic modulation were found in the as-coated Pt + γ'-Ni_3Al + γ-Ni coating shown in Fig. 1(c). Because the SAED patterns of these precipitates proved incompatible with the patterns of known phases such as γ and γ', the TEM specimen was tilted through a wide range of angles to obtain SAED patterns useful in determining its crystal structure. From these patterns [Figs. 1(e)–1(g)], we deduced that it has a hexagonal structure with lattice parameters $a = 0.473$ nm and $c = 2.565$ nm. This is consistent with the μ phase that is one of the TCP phases that sometimes form in Ni-based superalloys. The EDX spectrum of the μ phase (Fig. 2) indicates a refractory element rich compound containing the elements Re, W, and Co. The Pt content in γ'-Ni_3Al phase in the coating is about 16.5 at.% as measured by EDX. Electron diffraction reveals that there is no crystallographic relationship between the μ phase and the γ'-Ni_3Al. Figure 3(a) is SAED patterns of the μ phase along [010] zone axis. Reflections corresponding to both obverse (o) and reverse (r) forms of the rhombohedral cell[31] were present in SAED patterns. These patterns show that (001) twin domains exist in the μ phase. The twin domains parallel to (001) induce strong contrast modulation, as shown in Fig. 3(b).

Because of the low solubility of refractory elements in the γ'-Ni_3Al and γ-Ni phases in the coating, these refractory elements could have been trapped in a coating region during the heat treating used to diffuse the platinum into the nickel-based superalloy and later aluminizing, thereby leading to precipitation of the μ phase. TEM investigations of Pt + γ'-Ni_3Al + γ-Ni coatings showed that only the μ phase exists, no other TCP phases such as the σ, P, and R phases were found in specimens. TCP phases were formed in the $\gamma' + \gamma$ coating on René 5.[17] Similar results have been reported in Pt-aluminide

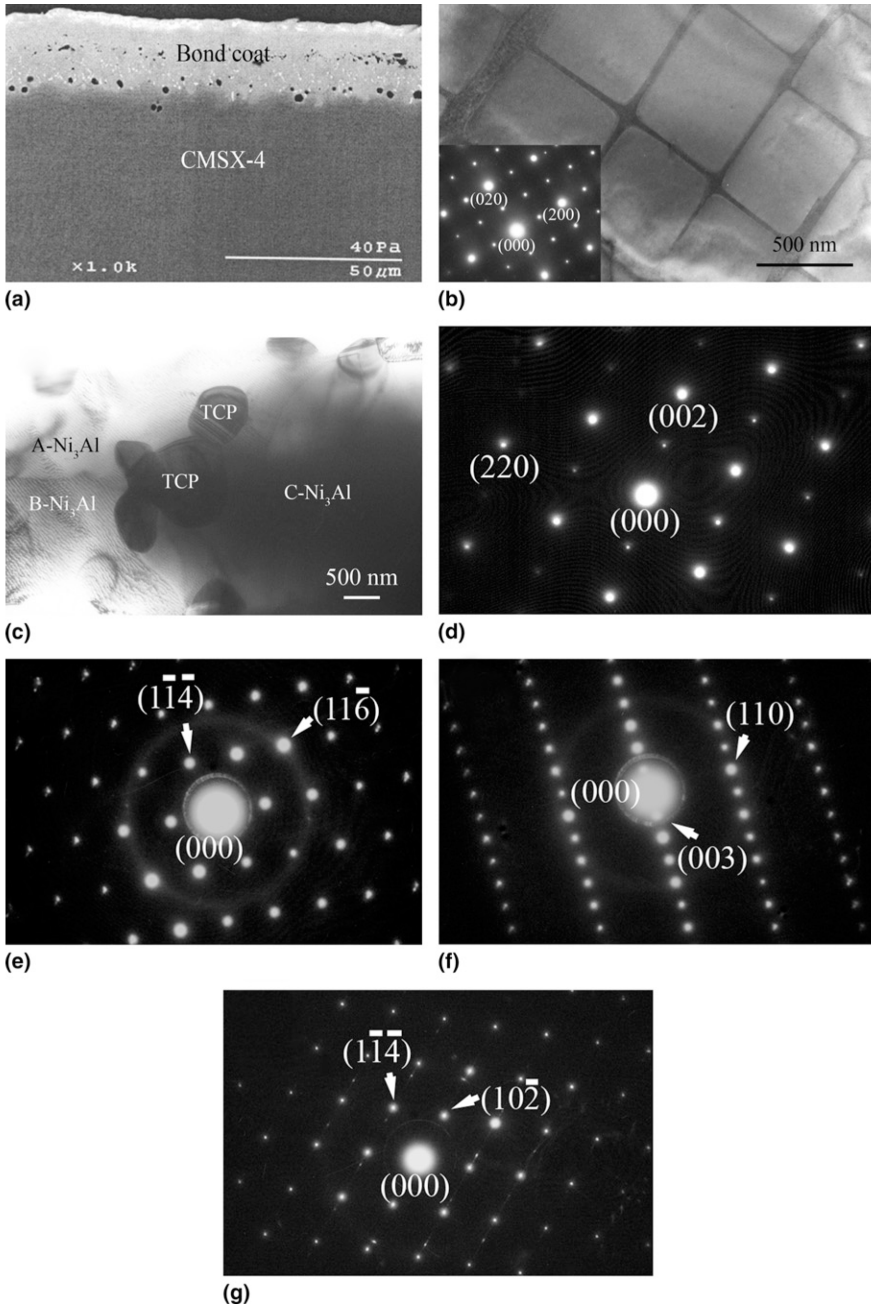

FIG. 1. (a) Cross-sectional scanning electron micrograph of the as-coated Pt + γ'-Ni_3Al + γ-Ni coating, (b) TEM image showing microstructure for CMSX-4 single-crystal superalloy substrate, the inset is SAED pattern of the γ'-Ni_3Al phase along the [001] zone axis, (c) cross-sectional TEM image of the as-coated Pt + γ'-Ni_3Al + γ-Ni coating, (d) SAED pattern of the region A revealing γ'-Ni_3Al phase along the [$1\bar{1}0$] zone axis, SAED patterns of the μ phase along the (e) [$\bar{5}\bar{1}\bar{1}$], (f) [$\bar{1}10$], and (g) [$\bar{2}2\bar{1}$] zone axes.

coating on CMSX-4 superalloy during the coating fabrication process, as well as during thermal mechanical fatigue.[27] Figure 2 shows that high levels of Re, W, and Co content were measured in the μ phase. Interestingly, Pt was absent in the μ phase according to EDX analysis.

The μ phase has been observed in Ni-based superalloys[7,24–26,32] and in the Co–W, Co–Mo, Fe–W, and Fe–Mo binary systems.[31,33,34] This precipitate exists at a 7:6 stoichiometry (e.g., Co_7W_6, Co_7Mo_6, Fe_7W_6, and Fe_7Mo_6). The crystal structure of the μ phase belongs to the $R\bar{3}m$ space group with lattice parameters ranging from 0.472 to 0.475 nm for a and 2.548 to 2.567 nm for c in the hexagonal system. Andersson[35] showed that the crystal structure of the μ phase could be derived from the β-tungsten structure using crystallographic operations such as translation, rotation, reflection, and intergrowth. The Co_7W_6 phase can be stacked alternatively and parallel to the (001) basal plane, in accordance with a

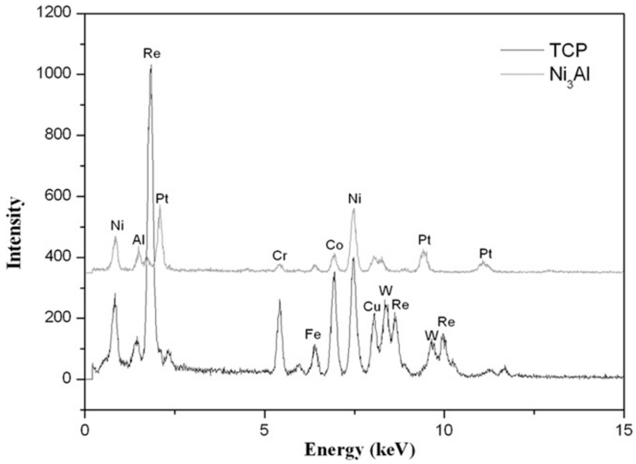

FIG. 2. EDX spectra of the μ and γ' phases in the coating.

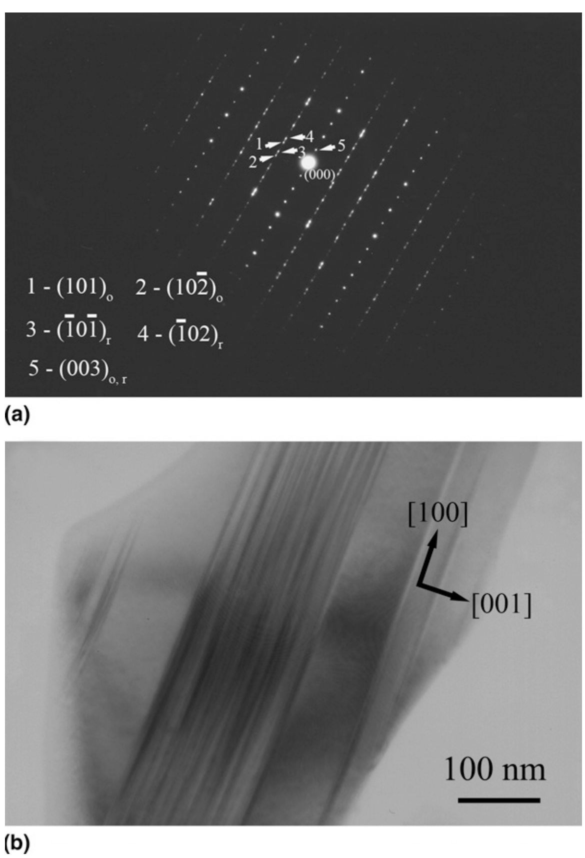

FIG. 3. (a) SAED patterns of the μ phase along the [010] zone axis showing twin domains parallel to (001) plane (o, obverse; r: reverse) and (b) TEM image of the μ phase showing strong contrast modulation due to twinning.

rhombohedral configuration. Twins will form by mistakes in the alternating intergrowth. SAED patterns in Fig. 3(a) confirm that (001) twinning exists in the μ phase in the present study. A twin domain parallel to (001) plane was generated by mirroring the lower part of the reverse cell onto the upper part of the obverse cell. Massive (001) twinning has been observed in the Co_7W_6, Co_7Mo_6, Fe_7W_6, and Fe_7Mo_6. The general reflection conditions for the μ phase require $-h + k + l = 3n$. $(00l)$ reflections will occur only at $l = 3n$. Therefore, the width of the twin domains is a multiple of $1/3c$. Figure 3(b) shows that the twin domains parallel to (001) induce strong contrast modulation. The randomly spaced domains are related by subunit cell mirror operation. Ab initio calculations[31] indicate that rather low energy is involved in the mirror operation; thus, the massive amount of twinning observed would not require high energy. A hexagonally close-packed (hcp) → $D0_{19}$ transformation was not found in the present work. Previous results on Co–W point to a displacive/diffusional Widmanstätten type of transformation, which is not simply based on a common ledge growth. The spatial arrangement of stacking faults and antiphase boundaries revealed the actual defect-formation mechanisms and disclosed important aspects of the phase transformation at an atomic level.[36]

In many cases, precipitation of TCP phases degrades mechanical properties by removing the refractory solid-solution strengthening elements from the constituent γ' and γ phases. The rodlike or platelike morphologies of the σ phase can severely reduce the rupture strength of superalloys at high temperature.[25] However, the effect of the μ phase on the mechanical properties is still unclear. Intergranular embrittlement has been observed when substantial amounts of acicular μ phase was present.[37] Simonetti and Caron[38] have investigated the role and behavior of the μ phase during deformation of a nickel-based, single-crystal superalloy. They showed that moderate amounts of acicular μ particles do not affect the tensile properties or the impact strength at room temperature. Moreover, the low-cycle fatigue properties of MC2 alloy at 650 and 950 °C are not affected by the presence of the μ phase. No detrimental effect on thermal mechanical fatigue lives (i.e., in terms of crack initiation and growth from TCP phases) were found in Pt-aluminide coatings on CMSX-4 superalloy.[27] Because TCP phases contain high refractory element contents, their precipitation can weaken the substrate even when they are present in blocky morphologies. For this reason, the TCP phases should be avoided. The detailed analysis of effects of the μ phase on the oxidation, corrosion, and thermal mechanical fatigue properties of γ'-Ni_3Al + γ-Ni coatings will be the subject of forthcoming papers.

IV. CONCLUSIONS

In summary, Pt-modified γ'-Ni_3Al + γ-Ni coatings on CMSX-4 single-crystal superalloys were successfully fabricated by Pt electroplating followed by a pack cementation process. TEM investigations of the cross-sectional Pt + γ'-Ni_3Al + γ-Ni/CMSX-4 coatings indicate that a blocky TCP, μ phase forms in the coating.

TEM analysis indicated that the μ phase has hexagonal crystal structure with lattice parameters $a = 0.473$ nm and $c = 2.565$ nm. The EDX spectrum reveals that the μ phase is rich in Re, W, and Co. Twin domains parallel to (001) have been observed in the μ phase.

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