



Effects of surface finish of single crystal superalloy substrate on cyclic thermal oxidation of its nanocrystalline coating



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ABSTRACT

Cycling oxidation behavior of the nanocrystalline coating on the N5 superalloy substrate of different surface finishing is investigated at 1050 °C. Results indicate that the surface finishing of the alloy substrate affects oxidation behavior of the sputtered nanocrystalline coating. After cyclic oxidation, oxide scale on the nanocrystalline coating rumples heavily and spalls off following a way of layer-by-layer in case that the alloy substrate is pretreated by sand blasting. However, it just roughens rather than rumples when the alloy substrate is pre-polished or ground by 2000# SiC paper and its spallation follows a way of bulk fracture.

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1. Introduction

Nanocrystalline coating [1–3] has recently attracted increasingly attention as a new bond coating (BC) for TBCs (thermal barrier coatings) system for its better compatibility with the alloy substrate than other traditional metallic coatings, such as aluminizing [4,5] and overlay NiCrAlY coatings [6,7]. Since its chemical composition is the same with the alloy substrate, any possible harmful effects induced by element inter-diffusion between the coating and the substrate can be avoided [2,3,8–15]. Besides, due to the columnar structure, thermal grown stresses transporting from oxide scale will be easily relaxed by deformation of the nanocrystalline coating, rather than by cracking of the oxide scale. Following our previous researches of compatibility between the nanocrystalline coating and many polycrystalline superalloy substrates [8–14], further efforts are now imposed on its application on single-crystal superalloys for high-temperature oxidation protection [1–3,15]. However, many challenges appear. For example, Wang et al. [2] reported that the nanocrystalline coating on a sand-blasted single-crystal superalloy rumples heavily after cyclic oxidation, which induced a typical ridge feature at surface of the oxide scale and even

minor spallation. Though this phenomenon in terms of rumpling of the nanocrystalline coating is similar to many platinum-modified nickel aluminide coatings [16–21], the acceptable explanations on rumpling of the latter are not absolutely appropriate for the former due to their different structures and chemical components. In this paper, it will be deduced that the co-effect of surface finishing of the alloy substrate and the columnar structure of the nanocrystalline coating accounts mainly to the final scale rumpling.

As an easy-processing surface finishing method, it has been reported that sand blasting (or shot-peening) can improve adhesive strength between substrate and its coatings by forming mechanical interlock, and decrease the corrosion rate as the process increased short diffusion path for Al or Cr [22,23]. Chen et al. [24] affirmed that sand blasting pre-treatment of alloy substrate enhanced the resistance to oxidation and spallation of the glass coatings by promoting formation of an alumina interlayer. Cruchley et al. [25] reported that shot-peening refined the microstructure at sub-surface, which promoted the selective oxidation of Cr, and in turn decreased oxidation rate of the chromia-forming superalloys. However, contradictory results still existed. Wu et al. [26] demonstrated that sand blasting influenced little on oxidation kinetics with the exception of suppressing DIR (diffusion induced recrystallization) of the K38 G alloy at high temperature. Richard et al. [27] explained that the increase in roughness by sand blasting induced an increase in residual stress near the interface and, consequently,

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Table 1
Composition of the single-crystal superalloy N5 (wt%).

Ni	Co	Cr	Mo	W	Ta	Al	Re	C	B	Y
Bal.	7.5	7.0	1.5	5.0	6.5	6.2	3.0	0.05	0.004	0.01

a decrease in adherence. Messe et al. [28], Chen et al. [29] and Foss et al. [30] also reported that sand-blasting induced plastic deformation and residual stress immediately under the alloy surface. According to Tolpygo et al. [31], it was shown that sand blasting resulted in about a tenfold increase of oxidation rate and extensive scale spallation during cyclic oxidation of the platinum-modified nickel–aluminide bond coatings on superalloys and, that the sand-blasting induced-impurities played a role of much importance in oxidation expediting. Thereafter, this research group carried out serials of detail investigations on scale rumpling of platinum-modified nickel–aluminide bond coatings. Results indicated that surface finishing is one of the main factors affecting scale rumpling, specifically speaking, sand blasting of the alloy substrate would not lead to surface rumpling while the polishing pretreatment did [32,33]. Xie et al. [34] argued that sand blasting of the (Ni, Pt)Al bond coat increased surface roughness which is directly related with the residual stress in thermal grown oxide and then the lifetime of the Y_2O_3 – ZrO_2 top coat of a TBC system.

In conclusion, sand blasting plays an important role on oxidation performance of alloys, e.g. oxidation rate and scale rumpling. This is rational since varying surface finishing results in a different topography and microstructure near surface. However, few reports have explained how sand blasting of the alloy substrates affects oxidation of the coatings on them. Obviously, surface roughness is one of the indirect causes that surface finishing of the alloy substrates affects performance of the coatings, but not the only one. Other factors such as residual compressive stress and microstructure refinement of the alloy substrate introduced by sand blasting affect as well the final oxidation behavior of some metallic coatings produced by PVD (Physical Vapor Deposition), as they affect growth of these PVD coatings. Differing from the previously published papers which focused on the effect of surface finishing on oxidation behavior of the alloy itself, this work was carried out with emphasis on the effect of surface finishing of alloy on oxidation behavior of the sputtering coating (one of a typical PVD coating) on it, rather than of the alloy itself. Their relationship as guidelines will be helpful as well in preparing other high-quality PVD coatings as the sputtering nanocrystalline coating.

2. Experimental method

The nominal composition of the single-crystal superalloy N5 is listed in Table 1. Cylindrical specimens of $\Phi 15 \times 2$ mm were cut from the superalloy bars and ground to 1000# SiC paper before final finishing. These samples are then divided into three groups. One group were sand-blasted with 100-mesh corundum particles at pressure of 0.2 MPa for 2 min (G1); one group were mechanically polished with 1 μ m diamond paste (G2); and the third group were ground to a final 2000# SiC paper (G3). After different surface finishing, these three groups of specimens were all degreased by an ultrasonic cleaner with acetone as substrates for sputtering the nanocrystalline coatings by magnetron sputtering. Before preparing this PVD coating, we have figured out that the sputtering rate is about 2.5 μ m per hour from a lot of preliminary tests. So the sputtering time was controlled for 11 h, making sure that a 25–30 μ m thick nanocrystalline coating was obtained. The sputtering target was a cast alloy sheet of $382 \times 128 \times 8$ mm in size and of the same composition with the single-crystal superalloy substrate. Atoms sputtered from this target deposited gradually onto the single-crystal alloy substrate to form the final nanocrystalline coating.

So the nanocrystalline coating should possess as well the same composition to the alloy substrate. Sputtering parameters were settled as follows: argon pressure was 0.2 Pa; sputtering current was 3.5 A; and substrate temperature was 200 °C. All the samples were cleaned in vacuum for half an hour to remove any oxides at surface before coating deposition and were rotated in the chamber during sputtering to ensure better uniformity.

Cyclic oxidation of these three groups of specimens was conducted by using a muffle furnace with an automatic device to drive specimens into and out of the furnace for 300 cycles in static air. In each cycle the specimens were exposed to the elevated temperature (1050 °C) for 1 h and then taken out from furnace and cooled in air for 15 min. There were five parallel specimens for each group. Mass change was recorded at interval of 10 cycles using an electron balance with a sensitivity of 0.01 mg.

Phase constituent was characterized by XRD (X'Pert PRO, PANalytical Co., Almelo, Holland, Cu K α radiation at 40 kV). The obtained X-ray diffraction patterns were recorded in 2θ range of 10–90° and, a step-scanning mode was employed with a step size of 0.02°. Morphologies and microstructures of the surface and cross section of the oxidized samples were examined by scanning electron microscopy (SEM, Inspect F 50, FEI Co., Hillsboro, Oregon) coupled with an energy dispersive spectrometer (EDS, X-Max, Oxford instruments Co., Oxford, UK). After different surface finishing processes, samples were immediately submitted to detect their surface roughness by a surface mapping microscope (Micro XAM-100, USA). A light spot of 3×3 mm in size was emitted onto the central of the tested samples, whose surface roughness is then obtained after analyzing the reflected light sign by the microscope. It is a nondestructive test method and the sample surface would not be contaminated during testing. So, the alloy substrates after roughness test can be deposited with the nanocrystalline coatings followed by the second measurement of surface roughness. After cyclic thermal oxidation, the third measurement of surface roughness was carried out for the same sample. In conclusion, surface roughness was measured third times for a single sample at three different stages: before coating deposition, after coating deposition and after cyclic thermal oxidation. For each group (G1–G3), three parallel samples were prepared.

3. Results

3.1. Cyclic oxidation kinetics and phase constituents

Fig. 1 shows cyclic oxidation kinetics at 1050 °C of the nanocrystalline coatings deposited on N5 superalloys with three different surface finishing. It is evident that all the three groups of nanocrystalline coatings provide high oxidation resistance independent on the surface finishing of the alloy substrate, as shown in Fig. 1(a). During the whole 300 cycles at 1050 °C, no obvious weight loss occurs, except that some mild weight fluctuation is observed after 100 cycles as indicated by arrows. This mild weight fluctuation may originate from small spallation at corners or from weighing errors. Among these three groups of nanocrystalline coatings, only minor difference in weight exists. In detail, nanocrystalline coating deposited on the polished superalloy substrate (G2) gains the lowest weight and G1 and G3 gain almost the same high weight after cyclic oxidation. However, considering that grinding and sand blasting roughen the specimen surface thus increase the real surface area exposed to atmosphere, the minor difference in mass gain

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