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ARTICLE

A New High Emissivity Coating on Ni-Based Superalloy Substrate

Wu Yongbin, Ma Xiaofei, Zhang Huazhen, Zhou Yang

Shanghai YS Information Technology Co., Ltd, Shanghai 200240, China

Abstract: In the present paper, we prepared a new high emissivity coating by a spray painting method. The coating contains amorphous borosilicate glass, $Mg_2B_2O_5$, $MoSi_2$ and SiB_4 and has the thickness of about 50 μm. Results show that the coating exhibits excellent thermal shock resistance (more than 100 thermal cycles from 950 °C to water temperature). The average emissivity of the coating is 0.905±0.024 at 950 °C and has a slight degradation after 100 thermal cycles.

Key words: coating; emissivity; thermal protection system; thermal shock resistance

With the development of inherent ductility, design flexibility and low maintenance cost metallic thermal protection systems (MTPS) for hypersonic flight vehicles[1,2], a research is initiated to provide a stable and high emissivity coating on superalloy substrate. And the coating is applied on metallic thermal protection system to decrease the surface temperature by radiation. However, the thermal shock resistance and emissivity remain a challenge to the current MTPS.

To increase thermal shock resistance of the coating on TPS panel, the coating on the MPTS need to be investigated. To our knowledge, due to borosilicate glasses with high viscosity, chemical passivity, and good ability for moistening different materials, the SiO_2-B_2O_3 system is a good basis for the elaboration of coatings[3]. Such coating possesses high emissivity and stability at peak heating under operational conditions, achieved by introduction of nonvolatile components into the borosilicate matrix. The nonvolatile additive is transition metal oxide including Cr_2O_3, CoO_x, Fe_2O_3[4], SiB_4, SiB_6[5], $MoSi_2$[6] and so on. With its high melting point, moderate density and excellent high temperature oxidation resistance, $MoSi_2$ becomes an important anti-oxidation candidate material of high temperature alloy or C/C composite. However, "Pesting" phenomenon occurs in $MoSi_2$ at 400~600 °C[7] due to vigorous oxidation. Therefore, $MoSi_2$ should be pulverized into powder, which will surely limit the application temperature range of $MoSi_2$. With thermodynamic compatibility of SiB_4 and $MoSi_2$ and excellent oxidation resistance of SiB_4, SiB_4 becomes one of major additives to improve the performance of $MoSi_2$. Moreover, SiB_4 has a high emissivity[8], so it could be widely deposited as high emissivity coating. Here we reported a new SiB_4-$MoSi_2$ composite coating by adding some toughening agent Mg_2B_2O using a spray painting method, which has high emissivity and excellent thermal shock resistance. This coating has the potential application in metallic thermal protection systems.

1 Experiment

Haynes 214 alloy was used as a substrate with the dimension of 100 mm×100 mm×0.07 mm. The substrate was polished with 200 mesh sand paper and ultrasonically cleaned in ethyl alcohol and acetone for 15 min and then dried. A coating slurry was prepared by ball-milling fine $Mg_2B_2O_5$ whisker and $Na_2Si_2O_3$, SiO_2, BC_4, $MoSi_2$ and SiB_4 particles for 8 h in distilled water, and then sprayed onto the substrate. Subsequently, the coating was dried in an oven at 100 °C for 1 h, and finally it was sintered at 850 °C for 1 h.

XRD was carried on a PANalytical X'Pert Pro X-ray diffractometer with Cu Kα radiation. The surface morphology of the coating was examined by scanning electron microscope

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Corresponding author: Wu Yongbin, Ph. D., Shanghai YS Information Technology Co., Ltd, Shanghai 200240, P. R. China, Tel: 0086-21-60912791, E-mail: yoopin@126.com

(SEM). The AFM surface topography was examined by the instrument NanoScope (R) Ⅲ, Version 5.3, Veeco instrument Ltd, with a scan rate of 0.5 Hz and scan range of 20 μm by tapping mode operation. The cross-sectional images of samples were examined by back-scattered electrons.

The spectral emissivity values of the coatings were measured on a self-made infrared radiometer of Harbin Institute of Technology, China. The spectral emissivity was determined by comparing the radiative fluxes between the samples and a blackbody under the same conditions. The radiation emitted by the front surface of sample was detected by a Fourier Transform Infrared spectrometer (JASCO FT/IR-6100), and its radiance was compared with the radiance of a blackbody radiator (Land R1500T Infrared, Dronfield, Sheffield S186DJ, England). The estimated uncertainly was less than 5%. Details of the emissivity measurement could be found in Ref.[8].

The thermal shock resistance was evaluated by a heating-cooling cycle test. The sample was heated up to 950 °C for 10 min, and then quenched by water for 5 min. This cycle was repeated for 100 times.

2 Results and Discussion

2.1 Phase compositions of the coating

The XRD patterns of the as-prepared coating of the Haynes 214 before and after 100 times thermal shock testing are shown in Fig.1. The XRD results indicate that the coating involves the amorphous borosilicate glass, $Mg_2B_2O_5$(JCPDS 15-0537), $MoSi_2$(JCPDS 41-0612), SiB_4(JCPDS 35-0777) and substrate. The amorphous glass borosilicate between 15°and 25° of 2θ is one of the phases. The thermal shock testing does not give rise to the phase composition change of the coating. This means that high temperature does not cause any chemical reaction among them.

2.2 Microstructure of the coating

The SEM images of the coating on Ni-base alloy are shown in Fig.2. The coating surface shows dense morphology without any crack, and exhibits the repeatable grooved finish, like V-shaped grooves, circular grooves, and pyramidal grooves which are commonly used to model the emissivity enhancement[1]. Meanwhile, SiB_4 and $MoSi_2$ surface is oxi-

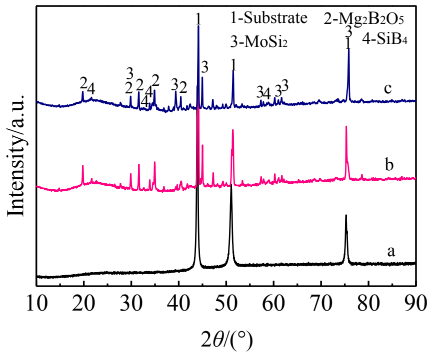

Fig.1 XRD patterns of the substrate (a), and the coating before (b) and after thermal shock (c)

dized to form dense SiO_2 film during sintering. And the fused borosilicate glass (main glass-forming constituents SiO_2 and B_2O_3) is less sticky and easy to flow into the crack, and thus self-heals the micro-cracks during sintering.

Fig.3 presents the cross-sectional image of the coating on Ni-base alloy. The coating is compact and has no crack within the coating thickness. The interface is clear, indicating there is no obvious inter-diffusion. The elemental distribution along coating thickness also confirms no inter-diffusion between substrate and the coating.

2.3 Thermal shock resistance tests

Thermal shock tests were carried out to detect the mechanical integrity and durability of the coatings at high temperature. Coating samples experienced the heat-up and cool-down cycle for 100 times. The surface morphologies of the coating before and after thermal shock testing are shown in Fig.4. After thermal shock testing for 100 cycles, the coating is still intact without flaking and burning, indicating that the coating has good thermal stability and thermal shock resistance.

Fig.5 is the comparison of AFM reports before and after thermal shock. The surface topography of the coating before thermal shock (Fig.4a) is very rough with mean surface roughness value (Ra) of 255.81 nm. Many surface protrusions are observed on the surface of the coating. In contrast, the mean surface roughness value (Ra) of the coating after thermal

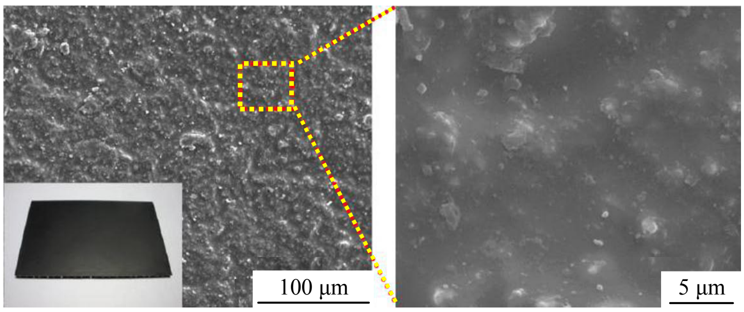

Fig.2 SEM images of the surfaces of the coatings