Ceramics International xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Oxidation resistance and mechanical properties of LaB_6-$MoSi_2$-SiC ceramic coating toughened by SiC nanowires

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ARTICLE INFO

Keywords:
Carbon/carbon composites
Coating
SiC nanowires
Oxidation resistance
Mechanical properties

ABSTRACT

To improve the high-temperature tolerance of carbon/carbon composites, a compact SiC-nanowires toughened LaB_6-$MoSi_2$-SiC/SiC (SiCnws-LMS/SiC) coating was designed and fabricated by combination of multiple methods including pack cementation, chemical vapor deposition and supersonic atmospheric plasma spraying. Isothermal oxidation results indicated that the mass loss of LMS/SiC coating decreased from 4.34 ± 0.28% to 1.12 ± 0.23% after oxidation for 200 h at 1773 K benefit from the addition of SiCnws. Absence of obvious cracks and voids in the coating after oxidation test indicated that the interfaces between various phases and SiCnws could obstruct the crack propagation by releasing the thermal stress in the coating. Meanwhile, after the introduction of SiCnws, the bonding strength and flexural strength of the coating were respectively increased by 54.54% and 59.77% compared to the LMS/SiC coating without SiCnws. The improved mechanical properties could be attributed to the pullout and bridging effects of SiCnws, which created multi-scaled reinforcements, thereby enhancing the load bearing capacity to increase the fracture toughness of the coating.

1. Introduction

The insatiable demand for next-generation engines with more harsh service environments (higher operating temperatures, hostile conditions and more) are required, which renders the menu of available materials vanishingly small [1,2]. Carbon/carbon (C/C) composites have received extensive attention from numerous researchers due to their excellent unique properties [3–5]. However, the higher oxidation rate of carbon material above 673 K in aerobic condition greatly damages carbon fibers in C/C matrix, resulting in the severe degradation of their mechanical properties [5–7]. The transport of gaseous species in C/C composites determines the oxidation process, forming some activation areas where both chemical reactions and gaseous diffusion occur. Therefore, the priority of protecting the C/C matrix is to block the contact between oxygen and matrix.

Intensive efforts are made to applying surface coatings to protect C/C composites against combustion and erosion at elevated temperatures [8–10]. Silicide ceramics are promising high-temperature materials for the long-term protection of C/C composites [11,12]. Over the past decade, SiC coating has been widely applied as a transition layer for protective systems due to its low coefficient of thermal expansion (CTE) and excellent compatibility with C/C composites [11–13]. However, the single-layer coating is difficult to achieve the long-term service capability at elevated temperatures, therefore the multilayer and multiphase coating systems are adopted by more researchers. In our previous studies [14–17], a multiphase LaB_6-$MoSi_2$-SiC (LMS) ceramic coating was prepared using an efficient supersonic atmospheric plasma spraying (SAPS), which exhibited a good oxidation resistance by forming a dense and adherent La-Si-O multiphase glass scale to block the diffusion of oxygen. Nevertheless, there are several challenges for LMS ceramic coating to be addressed: inherent brittleness of this ceramic coating, thermal mismatch between coating and substrate, spalling of La-Si-O glass scale under thermal cycle, which directly determine the protective capability of LMS ceramic coating at high temperatures.

To enhance the toughness and bonding strength of LMS ceramic coating and avoid the development of stress cracks, a practicable strategy is to incorporate the reinforcement materials such as nanoparticles [18], whiskers [13,19], nanowires [20] and nanotubes [21]. In the past few years, SiC-nanowires (abbreviated as "SiCnws") have been introduced into ceramic coating to increase coating toughness because of their superiority including smaller in size, better toughness and strength [20,22,23]. The SiCnws are barely damaged for flexibility when they cover the surface of carbon fiber. Its promoted antioxidant and mechanical properties make it more suitable for all kinds of the matrix materials. Nevertheless, the poor load-transfer between the

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https://doi.org/10.1016/j.ceramint.2018.06.045
Received 14 May 2018; Received in revised form 2 June 2018; Accepted 6 June 2018
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Please cite this article as: Wang, C., Ceramics International (2018), https://doi.org/10.1016/j.ceramint.2018.06.045

matrix and nanowires greatly limits the enhancement of SiCnws. Several efforts have been made to increase an interfacial contact area between the nanowires and the ceramic coatings [24,25]. In terms of the traditional SiCnws-toughened coatings, a discrete reticular framework of SiCnws with low aspect ratio was deposited on the coating matrix, which adverse to transferring load onto SiCnws. Conversely, establishing a continuous reticular nanowires structure can result in evenly deliver load over a large area and favor the releasing of thermal stress to improve the high-temperature stability of ceramic coatings [22,26]. Increasing of the aspect ratio of SiCnws is regarded as effective means for creating the continuous reticular framework in the coating.

In present work, the LMS/SiC coating toughened by continuous reticular SiCnws was prepared involving multiple techniques. Firstly, the C/C matrix was pre-oxidized to construct a porous surface structure, and the SiCnws were directly introduced into the porous C/C surface using chemical vapor deposition (CVD). Then the SiC coating was prepared on the SiCnws porous layer by pack cementation (PC), and the SiCnws was deposited on SiC coating through CVD method. In the end, the LMS coating was fabricated using SAPS technology to cover the outermost. The microstructure, phase composition, antioxygenic and mechanical properties of the coated specimens were investigated, along with the toughening mechanism of SiCnws in the coating.

2. Experimental procedure

2.1. Preparation of SiCnws-LMS/SiC coating

The C/C bulks selected for this study have a moderate density of 1.75 g cm^{-3} (manufactured by thermal gradient chemical vapor infiltration (TCVI) [3,27]), which were cut into small pieces with multiple sizes. After grinding with SiC sandpapers, they were cleaned with deionized water and alcohol ultrasonically in turn, then dried at 353 K for 4–8 h. Prior to the application of coating, these C/C specimens were first heat-treated at 1073 K with different time periods (2 min, 4 min, 6 min and 8 min). Afterwards, the SiC coating was fabricated on the pre-oxidized C/C surface using PC method with a mixed powder composed of 10 ~ 20 wt% graphite, 70 ~ 80 wt% Si and 5 ~ 10 wt% Al_2O_3, and the details are provided elsewhere [11,24,25]. Where-after, a high efficiency SAPS method was employed to manufacture the LMS coating with the composition of 60 ~ 70 wt% $MoSi_2$, 20 ~ 30 wt% SiC and 10 ~ 20 wt% LaB_6, and the specific spraying parameters are shown in Table 1. Before spraying, owing to the irregular shape and uneven particle size of commercially available powders, they must be agglomerated to near-spherical shape with the size range of 30 ~ 100 µm using spray granulation method. Spray-drying suspensions were obtained by blending $MoSi_2$, SiC and LaB_6 powders with 2 ~ 4 wt% bonding agent (polyvinyl alcohol (PVA)). The suspension was ball-milled for 6 ~ 8 h and atomized, then the collected granules were sieved for spraying.

For the synthesis of SiCnws in this study, they grew in situ on the pre-oxidized C/C surface and PC-SiC coating using CVD with the precursor powder composition of 50 ~ 60 wt% SiO_2, 10 ~ 20 wt% Si, 20 ~ 30 wt% graphite and 5 ~ 10 wt% ferrocene. These powders were stirred with a mechanical ball-milling for 2 ~ 4 h, and then spread on the bottom of graphite crucible. The specimens were suspended over the mixed powders in the crucible with graphite lid, and then they were heated to 1873 K for 2 ~ 3 h in the furnace with vacuum and argon shielding. A schematic diagram of SiCnws-toughened LMS/SiC coating (abbreviated as SiCnws-LMS/SiC) is shown in Fig. 1.

Table 1
Detailed of spraying parameters for SAPS-LMS coating.

Parameters	Numerical range
Operating current (A)	400–450
Operating voltage (V)	100–150
Primary gas Ar (L min^{-1})	70–80
Carrier gas Ar (L min^{-1})	5–15
Second gas H_2 (L min^{-1})	5–10
Nozzle diameter (mm)	5.5
Spraying distance (mm)	110–130
Powder feed rate (r min^{-1})	4.0–4.5

2.2. Oxidation tests

Non-isothermal oxidation behavior of the specimens with LMS/SiC coating and SiCnws-LMS/SiC coating was measured by the thermogravimetric (TG) analyzer (measuring range showed in Table 2) in an atmospheric environment, and the heating rate was controlled at 5 K min^{-1}.

Isothermal oxidation test was performed in the 1773 K constant high-temperature resistance furnace (measuring range showed in Table 2). The furnace was firstly heated to a predetermined temperature at the heating rate of 5 K min^{-1}, and then the specimens were directly placed into the furnace chamber. After oxidation to a certain time, the specimens were removed from the furnace and weighed with an electronic analytical balance (measuring range showed in Table 2). Average value from three separate specimens for each group under the similar condition was applied to increase data accuracy. The mass change percentage (Δw_1, %), per unit area mass change (Δw_2, mg cm^{-2}) and per unit area mass change rate (Δw_3, mg cm^{-2} h^{-1}) of the specimens after oxidation was defined by the following Eqs. (1)–(3):

$$\Delta w_1 = \frac{m_0 - m_t}{m_0} \times 100\% \tag{1}$$

$$\Delta w_2 = \frac{m_0 - m_t}{S} \tag{2}$$

$$\Delta w_3 = \frac{m_0 - m_t}{S \cdot t} \tag{3}$$

where m_0 is the initial mass of the specimen before oxidation, m_t is the mass of the specimen after oxidation. S and t represent the total surface area of the specimen and sustained oxidation time, respectively.

2.3. Mechanical properties

Flexural strength of the specimens (30 mm × 5 mm × 3 mm) was evaluated using the three-point bending test (universal testing machine, measuring range showed in Table 2), and the loading rate was kept at 0.5 mm min^{-1}. The final result after testing was an average of four separate specimens. Flexural strength (σ_f) was calculated by following Eq. (4):

$$\sigma_f = \frac{3P_f \cdot s}{2bw^2} \tag{4}$$

where P_f is the maximum breaking load, s is the distance between the fulcrum (support span), b is the width of the specimen, w is the height of the specimen.

A tensile test was employed to measure the bonding strength between LMS coating and SiC coating using universal testing machine (measuring range showed in Table 2) according to the ASTM-C633 standard [28]. Detailed procedures have been reported elsewhere [9,14]. Five specimens were selected for each group to increase the accuracy of the experimental data.

2.4. Characterization

Phase composition and crystalline structures of the coated specimens were characterized using X-ray diffraction (XRD). Raman spectrometer (RMS) and transmission electron microscopy (TEM) were applied to analyze the nanostructured SiCnws. Scanning electron microscopy (SEM) was exploited to characterize the microstructure and

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