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Formation and oxidation resistance of a new YSZ modified silicide coating on Mo-based alloy



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HIGHLIGHTS

- A new YSZ modified Si-Mo coating was
 designed and prepared on Mo-based
- designed and prepared on Mo-based alloy by slurry reaction sintering method.Microstructure and oxidation behavior
- Microstructure and oxidation behavior of the coating with different amounts of YSZ were studied.
- The Si-Mo-10YSZ remained intact after oxidation at 1725 °C for 22 h and the mass gain was 0.96 mg/cm².
- The excellent performance was attributed to the dense SiO₂-ZrO₂-ZrSiO₄ scale.

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ABSTRACT

A new YSZ (yttria stabilized zirconia) modified silicide coating was prepared on Mo-based alloy through a twostep process, including spraying of Si-Mo-YSZ slurry followed by reaction sintering. Microstructure and oxidation behavior of the coatings with different YSZ content (0, 5, 10 and 15%) were studied. The coating consists of a MoSi₂-ZrSi₂-SiO₂ outer layer and a MoSi₂ inner layer. The 10% YSZ modified coating possessed the best oxidation protection at ultra-high temperatures and, for the coating, the mass gain was 0.96 mg/cm² after being exposed at 1725 °C in air for 22 h, whereas the uncoated Mo-based alloy completely ablated for 0.35 h. The excellent oxidation resistance of the silicide coating was attributed to the compact and self-healing SiO₂ protective scale, which contained the dispersed ZrO₂ and ZrSiO₄.

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1. Introduction

Of high melting point (2620 °C), low thermal expansion coefficient, moderate density (10.23 g/cm³), good mechanical properties, and excellent thermal shock as well as thermal fatigue resistance, molybde-num and its alloys have become important structural materials for applications in high temperatures (1000–2000 °C) [1–4]. For instance,

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they are widely applied in aerospace and nuclear industries as nozzles and nose cone of rocket jets, leading edge and rudder of aircrafts and core materials of nuclear reactors [5, 6]. However, oxidation resistance of molybdenum is poor in high-temperature aerobic environment. At about 200 °C, molybdenum begins to form a thin oxide layer. From 500 to 700 °C, weight gain of molybdenum obeys linear rule. Above 700 °C, catastrophic pulverization occurred in molybdenum due to eutectic reaction taking place between MoO₂ and MoO₃ [7–10]. It is imperative to improve oxidation resistance of molybdenum and its alloys before they can be considered for high-temperature applications. Two approaches have been tested in the past: one is adding alloying elements to alloy with refractory metals to enhance oxidation resistance [11–13]. However, as the low content of modified elements cannot maintain a continuous dense oxidation protective layer, the new refractory alloys are difficult to be used at high temperature for a long time. Meanwhile, excessive addition easily leads to a sharp deterioration in the mechanical properties of alloys. Another is to prepare a high temperature oxidation-resistant coating on metal surface [14, 15]. Without changing the composition of the base alloy, or affecting its inherent performance, this method is effective in improving the oxidation resistance at high temperature.

Heat-resistant alloy coatings [16], aluminide coatings [16, 17] and silicide coatings [18, 19] have been proposed and examined for molybdenum and its alloys. Among those, silicide coating represented by MoSi₂ is one of the best candidates due to its excellent high temperature oxidation resistance and matched coefficient of thermal expansion [20–23]. With rapid development of aerospace and nuclear technology, working temperature of hot-end components has been increased to over 1500 °C, and even 1800 °C at short service time [5], while the service temperature of pure MoSi₂ coating cannot exceed 1650 °C as the melting point of SiO₂ film is 1670 °C (tridymite) to 1710 °C (cristobalite) [24, 25]. Addition of high melting point substances (such as WSi₂, Si₃N₄, and Zr) into MoSi₂ is therefore considered as an appropriate way to improve its high temperature oxidation resistance [15, 26].

Featured with high melting point (2690 °C), stable chemical properties, low oxygen permeation rate and excellent ablation resistance, yttria stabilized zirconia (YSZ) could be a potential addition for silicide coating [27–32] if their thermal expansion mismatch can be tailored (CTE_{YSZ} = 10.8×10^{-6} /°C, vs CTE_{Mo} = 4.9×10^{-6} /°C) [33, 34]. On the other hand, zirconia and Si can in-situ produce ZrSi₂ and SiO₂ at high temperature [35, 36] with close thermal expansion (CTE_{ZrSi2} = 8.5×10^{-6} /°C, vs CTE_{MoSi2} = 8.1×10^{-6} /°C) [37, 38]. In the meantime, SiO₂ can further reduce the thermal expansion coefficient of coating (CTE_{SiO2} = 0.55×10^{-6} /°C) [39] with good self-healing properties. Therefore, through reasonable design and preparation, YSZ is expected to improve high temperature oxidation resistance of MoSi₂ coating [40, 41]. Based on the mentioned discussion, we have come into a decision to design and test a new type of Si-Mo-YSZ coating. To our knowledge, no such work has ever been reported yet.

In the present work, a simple, efficient and low-cost slurry sintering was selected from numerous preparation methods for YSZ modified Si-Mo coating. Samples were prepared through a two-step process including spraying of Si-Mo-YSZ slurry on Mo-based alloy, followed by reaction sintering [42]. Morphology and properties of silicide coatings with different YSZ content were analyzed. Oxidation behavior of YSZ modified silicide coatings was investigated up to 1725 °C.

2. Experimental procedures

2.1. Substrate materials

Titanium-Zirconium-Molybdenum (TZM) alloy of nominal composition Mo-0.5Ti-0.1Zr-0.02C (wt%) was chosen as substrate. This TZM alloy was prepared by vacuum arc consolidation (ZKY-10A, Zhuzhou Chikoyuan mechanical and electrical co., Ltd.) method where ingot undergone at least 5 times re-melt to ensure composition uniformity. Plates of 60 mm \times 5 mm \times 1 mm was taken from the ingot via wire cutting method. Upon grinding by silicon carbide paper of 200-grit, 400grit and 600-grit, the plates were cleaned in an ultrasonic acetone bath and dried at 80 °C for 1 h for later use (as substrate).

2.2. Coating process

A two-step method was adopted to prepare silicide coating on the surface of molybdenum alloy, as shown in Fig. 1. First, slurry of composition 60Si-(40-x) Mo-xYSZ (wt%), where x was 0%, 5%, 10% and 15% was prepared from mixture of YSZ, Si and Mo powders at average particle size of about 100 nm, 1 µm and 3 µm, with purity over 99%, respectively. Small amount of halide(NH₄F), lyvinyl butyral (PVB) and SiO₂ powders (purity more than 99% and averaged particle size of 2 µm, 5 μ m, and 0.1 μ m, respectively) were added into the mixture, and were ground in a planetary ball mill, operated at 400 r/min, using ethyl alcohol as a solvent, for 6 h. The mixed slurry had an irregular shape (Fig. 2a). The particle size was not uniform with an average of 1.276 µm measured by laser particle size analyzer (Mastersizer Microplus Ver. 2.19, c/o Malvern Instruments Ltd.) (Fig. 2b). The slurry was sprayed evenly on the surface of the TZM alloy specimen by an air compressor and spray gun (EW15008, Hongwuhuan Group Co., Ltd.). The spray parameters used in this experiment were: slurry viscosity of $3.8-4.0 \times 10^{-2}$ Pa·s, spraying distance of 20–30 cm, air pressure supplied to the spray gun of 0.8 MPa and spray gun nozzle with a diameter of 0.5 mm. Secondly, after being dried at 110 °C for 1 h, the sample with the slurry on the surface was suspended in a molybdenum alloy crucible, then placed in a vacuum sintering furnace (WZK-1800, Changsha Wande Powder Metallurgy Equipment Co., Ltd.). The temperature was increased from room temperature to 500 °C at a rate of 5 °C/min, and held for 1 h to remove water vapor and PVB. Then, high purity argon (99.999 wt%) was purged. Afterwards, the temperature was raised to 1450 °C and maintained for 1 h under argon, then the furnace was slowly cooled down to prepare the final product. An additional halide activated pack cementation [43] can be carried out to further ensure the complete silicidation of the coating.

2.3. Oxidation test

The isothermal oxidation behavior of the specimens was tested at 1725 °C in the air using a home-made oxidation equipment, equipped with real-time display and automatic data recording. The sample was heated to 1725 °C at a heating rate of 500 °C/min, then was oxidized at the constant temperature and timing was started. The temperature error was ± 8 °C, and it was weighed every 1 h. The mass change of the specimens was measured by an electronic analytical balance with sensitivity of ± 0.1 mg. For each slurry composition, five samples were measured, where the average value was used. At the early stage of oxidation, a layer of glassy oxide film was rapidly formed on the surface of the coating. At the later period, when the coated sample have shown obvious failures such as the appearance of smoke, then it was removed and the time was recorded.

2.4. Microstructure characterization

The as-coated and oxidized specimens were characterized by the scanning electron microscopy (SEM) (FEI Sirion 200, USA) with an energy dispersive spectrometer (EDS), X-ray diffraction (XRD) (Model D/Max 2500PC Rigaku, Japan), and electron-probe micro-analysis (EPMA) (Model JEOL JXA-8230, Japan) with a wavelength dispersive spectroscopy (WDS). The microstructure was characterized using a high-resolution transmission electron microscope (HRTEM, FEI Tecnai G2 F20, America). The roughness values of the coatings were analyzed using a 3D laser scanning confocal microscope (Carl Zeiss LSM700, Germany).

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