



## Hot corrosion behavior of Ba<sub>2</sub>DyAlO<sub>5</sub> exposed to calcium-magnesium-alumina-silicate at 1300 °C and 1350 °C

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### ABSTRACT

Calcium-magnesium-alumina-silicate (CMAS) attack has been considered as one of the significant failure mechanisms for thermal barrier coatings (TBCs). In this study, CMAS corrosion behavior of Ba<sub>2</sub>DyAlO<sub>5</sub> is investigated at 1300 °C and 1350 °C for 0.5 h, 4 h, 12 h and 24 h. The results indicated that Ba<sub>2</sub>DyAlO<sub>5</sub> had high resistance to CMAS infiltration for prolonged durations (24 h), ascribed to the rapid formation of crystallization phases by chemical reaction between Ba<sub>2</sub>DyAlO<sub>5</sub> and CMAS. The corrosion products with a main constitution of Ca<sub>2</sub>Dy<sub>8</sub>(SiO<sub>4</sub>)<sub>6</sub>O<sub>2</sub>, Ba<sub>1.31</sub>Ca<sub>0.69</sub>SiO<sub>4</sub>, BaAl<sub>2</sub>O<sub>4</sub> and Dy<sub>2</sub>O<sub>3</sub> phases formed a sealing layer on the sample surfaces, which can effectively suppress further penetration of the molten CMAS. The associated mechanism was discussed.

### 1. Introduction

Thermal barrier coatings (TBCs) enable the hot parts of aircraft gas turbine engines to operate at higher temperatures, which effectively enhance the operating efficiency of the engines [1–4]. However, the increasing demand for higher operating temperature induces new issues. At elevated temperatures, the siliceous debris such as volcanic ash, fly ash, dust, sand and runway debris ingested into engines with the intake air poses a severe threat to the life of TBCs [5–7]. The debris mainly consists of CaO, MgO, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>, and thus they are collectively expressed as CMAS. At high temperatures (above 1200 °C), CMAS melts and penetrates into the TBC through the cracks and pores by a capillary action, resulting in damaging the integrity of the coating [9–11]. As a result, a premature spalling of the coatings occurs during service.

At present, the most successful ceramic top coat material for TBCs is yttria partially stabilized zirconia (YSZ), which is usually prepared by air plasma spray (APS), electron beam physical vapor deposition (EB-PVD) and plasma spray physical vapor deposition (PS-PVD) [12–14]. Substantial reports have revealed that YSZ TBCs are particularly vulnerable to CMAS attack. Related degradation mechanisms refer to two aspects, i.e. thermo-chemical and thermal-mechanical mechanisms. Molten CMAS readily dissolves YSZ grains, causing the enrichment of

Y<sup>3+</sup> and Zr<sup>4+</sup> in the molten CMAS. The re-precipitated ZrO<sub>2</sub> grains are depleted in Y<sub>2</sub>O<sub>3</sub> due to the relatively low solubility of Zr<sup>4+</sup> in CMAS melt compared with Y<sup>3+</sup> [15–17]. Moreover, the Y-lean ZrO<sub>2</sub> grains suffer martensitic phase transformation during thermal cycling process accompanied with a destructive volume expansion that causes seriously damage to TBCs. The thermo-mechanical mechanism has also been revealed. The CMAS-impregnated TBCs exhibit significant low strain-tolerance, leading to an increasing thermal misfit stress during thermal cycling [16,18,19]. When the stress is accumulated to a certain degree, the TBCs would spall prematurely. Owing to inevitable siliceous debris and ever growing demands for higher engine operating temperatures, CMAS attack to TBCs is becoming an extremely decisive issue in the development of the next-generation gas-turbine engines.

In recent decades, the TBCs degradation induced by CMAS attack is still a huge issue. Relentless efforts have been done to alleviate degradation by CMAS attack. They include introducing a protective layer on TBC surfaces and modifying the chemistry composition of YSZ [20–23]. Additionally, alternate materials have also been explored. Krämer et al. firstly revealed that Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> are highly resistant to CMAS attack [8]. CMAS melt rapidly dissolves Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> grains, and concurrently precipitates an apatite phase based on Ca<sub>2</sub>Gd<sub>8</sub>(SiO<sub>4</sub>)<sub>6</sub>O<sub>2</sub> and ZrO<sub>2</sub> with Gd and Ca in a solid solution. These crystallized products with high melting temperature form a dense reaction layer, blocking

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CMAS further penetration. Other TBC candidates such as  $\text{La}_2\text{Ce}_2\text{O}_7$ ,  $\text{La}_2(\text{Zr}_{0.7}\text{Ce}_{0.3})_2\text{O}_7$  and  $\text{GdPO}_4$  are also found to have excellent resistance to CMAS attack [10,24,25]. The rationale behind the desirable property is similar, mainly attributed to the formation of apatite phase with the help of enough  $\text{RE}_2\text{O}_3$  contents.

$\text{Ba}_2\text{DyAlO}_5$  has been proposed as a prospective TBC candidate material. The thermal conductivities of  $\text{Ba}_2\text{DyAlO}_5$  have achieved  $1.1 \text{ W/m}\cdot\text{K}$ , which is smaller than half of 7YSZ. Meanwhile, the thermal expansion coefficient of  $\text{Ba}_2\text{DyAlO}_5$  is close to  $12.0 \times 10^{-6} \text{ K}^{-1}$  at  $1300^\circ\text{C}$ , which is higher than that of 7YSZ ( $11.0 \times 10^{-6} \text{ K}^{-1}$ ).  $\text{Ba}_2\text{DyAlO}_5$  also exhibits good chemical compatibility with  $\text{Al}_2\text{O}_3$  from the thermally grown oxide layer and excellent CMAS resistance [26–29]. In addition, it is predicted that  $\text{Ba}_2\text{DyAlO}_5$  has excellent CMAS resistance due to enough  $\text{RE}_2\text{O}_3$  content. Higher operating temperatures are long term goals and  $1300^\circ\text{C}$  is the representative of surface temperature targets for further engines design. However, there are lack of reports pertaining to the CMAS corrosion behavior of  $\text{Ba}_2\text{DyAlO}_5$  material at higher temperatures ( $1300^\circ\text{C}$  and  $1350^\circ\text{C}$ ) and the evolution process of CMAS penetration. In this study,  $\text{Ba}_2\text{DyAlO}_5$  pellets, instead of actual TBCs, are used to perform CMAS corrosion studies at  $1300^\circ\text{C}$  and  $1350^\circ\text{C}$  for different heat treatment durations (0.5 h, 4 h, 12 h and 24 h). The researches aim to explore CMAS corrosion mechanism and the time-dependent reaction kinetics.

## 2. Experimental procedure

$\text{Ba}_2\text{DyAlO}_5$  powders were synthesized by a solid state reaction method, selecting  $\text{BaCO}_3$  (Real&Lead Chemical Co., Ltd., China; purity  $\geq 99.99\%$ ),  $\text{Al}_2\text{O}_3$  (Tianjin Jiangtian Chemical Technology Co., Ltd., China; purity  $\geq 99.99\%$ ) and  $\text{Dy}_2\text{O}_3$  (Beijing HWRK Chem Co., Ltd., China; purity  $\geq 99.99\%$ ) powders as reactants. Before weighing, dysprosium oxide powders were calcined at  $900^\circ\text{C}$  for 4 h to remove absorbed moisture. The appropriate mole ratio reactants were suspended in alcohol and then mixed uniformly using a planetary ball mill (WL-2L, China), followed by drying at  $80^\circ\text{C}$  for 12 h. The dried powders were pre-calcined at  $1200^\circ\text{C}$  for 10 h and then sintered at  $1500^\circ\text{C}$  until a single-phase  $\text{Ba}_2\text{DyAlO}_5$  was synthesized. Subsequently, the pure  $\text{Ba}_2\text{DyAlO}_5$  was grinded and sieved to acquire homogeneous powders. The obtained powders were cold pressed at 300 MPa and then sintered at  $1500^\circ\text{C}$  for 10 h to obtain pellets. Prior to CMAS corrosion tests, the pellets were ground employing 400, 800 and 1000 grit SiC abrasive papers, and then polished.

Laboratory CMAS powders with a composition of  $22\text{CaO}-19\text{MgO}-14\text{AlO}_{1.5}-45\text{SiO}_2$  in mole percent of single cation oxide formula units were used in this study. All the oxides (Tianjin Jiangtian Chemical Technology Co., Ltd., China; purity  $\geq 99.99\%$ ) were taken in corresponding mole ratio and dissolved in deionized water, followed by sufficiently ball-milling for 12 h. The obtained suspension was thoroughly dried at  $150^\circ\text{C}$  for 8 h, followed by calcining at  $1250^\circ\text{C}$  for 4 h. The products were grinded and sieved to achieve an appropriate particle size. The CMAS powders were dispersed uniformly on the surface of as-sintered  $\text{Ba}_2\text{DyAlO}_5$  pellets with a concentration of  $20 \text{ mg/cm}^2$ . CMAS corrosion experiments were performed isothermally in a box electric resistance furnace at  $1300^\circ\text{C}$ ,  $1350^\circ\text{C}$  for 0.5 h, 4 h, 12 h and 24 h.

X-ray diffraction (XRD; Rigaku Diffractometer, Tokyo, Japan) with CuK $\alpha$  radiation was employed for phase constitution analysis. Data was digitally recorded in a continuous scan within the scope of angle ( $2\theta$ ) from  $10^\circ$  to  $80^\circ$  with a scanning rate of  $0.2^\circ/\text{s}$ . Differential scanning calorimetry (DSC; STA 449C, NETZSCH Ltd., Germany) analysis was carried out from room temperature to  $1400^\circ\text{C}$  to investigate the chemical reaction between CMAS and  $\text{Ba}_2\text{DyAlO}_5$ . The set heating and cooling rate was  $10^\circ\text{C}/\text{min}$ . The microstructure and chemical compositions of corroded samples were conducted using a scanning electron microscope (SEM; TDCLS4800, Hitachi Ltd., Japan) equipped with energy dispersive spectroscopic (EDS, IE 350).

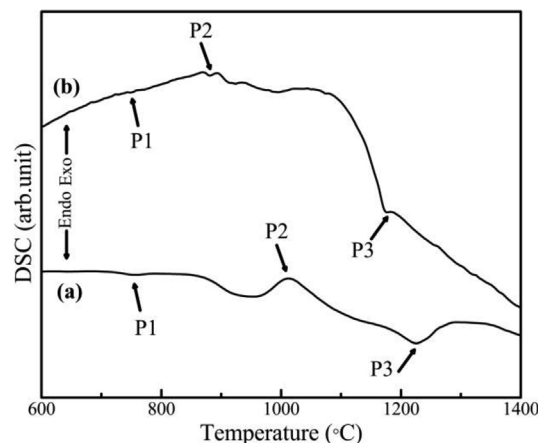


Fig. 1. The DSC patterns of CMAS (a) and 50 wt% CMAS-50 wt%  $\text{Ba}_2\text{DyAlO}_5$  mixtures (b).

## 3. Results and discussion

### 3.1. Investigation of the chemical reaction between CMAS and $\text{Ba}_2\text{DyAlO}_5$ by DSC analysis

Fig. 1a shows the DSC curve of CMAS powders. As shown in Fig. 1a, a small endothermic peak (P1) at about  $756^\circ\text{C}$  could be found, which could be attributed to glass transition of CMAS. The exothermic peak (P2) is observed at about  $1012^\circ\text{C}$ , indicating the crystallization of CMAS powders. There is a noticeable endothermic peak (P3) at about  $1226^\circ\text{C}$ , suggesting that at this temperature range CMAS begins to melt. This result conforms to the study by Kramer et al. [30]. To investigate

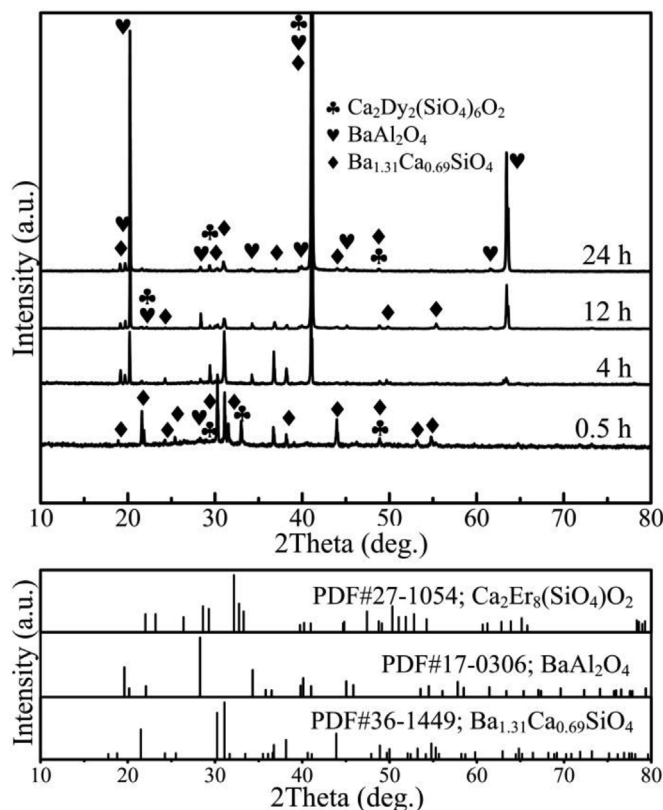


Fig. 2. XRD patterns of  $\text{Ba}_2\text{DyAlO}_5$  pellets with CMAS deposits at  $1300^\circ\text{C}$  for 0.5 h, 4 h, 12 h and 24 h, and the standard XRD PDF cards of  $\text{Ca}_2\text{Er}_8(\text{SiO}_4)_6\text{O}_2$ ,  $\text{Ba}_{1.31}\text{Ca}_{0.69}\text{SiO}_4$  and  $\text{BaAl}_2\text{O}_4$ .

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