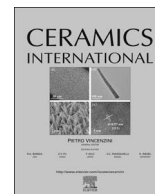




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Corrosion behavior of volcanic ash on sintered mullite for environmental barrier coatings

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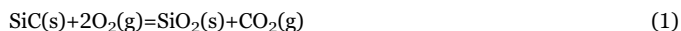
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ABSTRACT

The high-temperature corrosion behavior of volcanic ash (VA) in attacking sintered mullite was investigated, and the corrosion resistibility of mullite environmental barrier coatings (EBCs) to VA was predicted. Sintered mullite specimens were prepared by using the spark plasma sintering method. These specimens were subjected to a hot corrosive environment—molten Icelandic VA at 1400 °C—for three different duration times (2, 12, and 48 h). The microstructure and phase of the specimens were analyzed by using a scanning electron microscope equipped with an accessory system for energy dispersive spectroscopy and X-ray diffraction. In addition, *in-situ* high-temperature X-ray diffraction was carried out to identify the dynamics of phase evaluation in the volcanic ash and mullite mixture powders. Results show that a reaction layer was generated and continuously dissolved into the melted volcanic ash. The primary incursive component is iron; however, a minimal amount of sodium plays a more important role in disintegrating sintered mullite.

1. Introduction

The improvement of gas turbine inlet temperature is a key factor in increasing the fuel efficiency and reducing the carbon emissions of a gas turbine. Such improvement is dependent on the development of advanced materials [1–3]. Given the high limit point of temperature capability, non-oxide silicon-based ceramics such as Si₃N₄ and SiC have been investigated extensively as a potential structural material for hot gas parts for next-generation gas turbines [4,5]. However, numerous investigations have indicated that the disadvantageous factor of Si₃N₄ and SiC as it applies to gas turbines is that these ceramics lose observable weight in a combustion environment. Owing to the presence of oxygen and high-temperature water vapor in a combustion environment, SiC creates volatile silicon hydroxide [Si(OH)₄] via reactions (1) and (2) with a recession rate of close to 1 μm/h when the temperature rises to 1300 °C.



To solve this problem, a protective layer-called environmental barrier coating (EBC) is necessary [6–10]. Qualified EBC candidate materials require some fundamental chemical properties, such as a high melting point, low thermal conductivity, low oxygen diffusion, favorable chemical stability, and thermodynamic compatibility with the substrate. In addition, EBCs require a highly dense architecture to ensure that all the paths between the substrate and the oxygen/steam are blocked. Therefore, an appropriate coefficient of thermal expansion (CTE) may be the most critical property for EBC candidate materials. On the basis of the abovementioned requirements, mullite (3Al₂O₃·2SiO₂) [11] has attracted the most interest as a commercial protective layer for non-oxide silicon-based ceramics because of its reasonable price, excellent adherence, chemical compatibility, and appropriate CTE with SiC and Si₃N₄ substructures.

Generally, research on the degradation of EBCs focuses on the corrosion behavior and resistibility of high-temperature water vapor and oxygen. However, since the eruption of the Icelandic volcano, which caused economic losses of more than 2 billion dollars, investigators have found that the more serious threats to the stability of EBCs probably come from sand, volcanic ash (VA), and other siliceous matter

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[12,13]. One reason is that sand and VA may be considered foreign object debris that affects EBCs. A particle that can enter a combustion gas stream with a velocity of 600 m/s can attack the EBC while the turbine machinery is operating, thereby causing radial cracks and damage in the EBCs [14]. Another report [15] discussed for the first time the phenomenon of VA attaching to aircraft engines and indicated that, compared with the effect of mechanical damage, more serious degradation of EBCs may come from chemical corrosion. According to literature [16] that further investigated the corrosion behavior between thermal barrier coating (TBC) degradation and dust from the Middle East, the core composition of the attachment is calcium-magnesium-alumino-silicate (CMAS). According to the literature [17], serious degeneration as a result of CMAS is observed not only for TBCs but also for EBCs whose composition is BSAS ($\text{Ba}_{1-x}\text{Sr}_x\text{Al}_2\text{Si}_2\text{O}_8$).

Given that the core composition of sand and VA is defined as CMAS, and CMAS is relatively easy to obtain and analyze under laboratory conditions, most studies use CMAS in place of natural sand and VA as a corrosive for EBCs. However, because scientists who use real VA to attack TBCs [18,19] have indicated that iron (Fe), sodium (Na), and manganese (Mn) components, which are not included in CMAS, also penetrated the substructure, the use of natural sand or VA as a corrosive for EBCs can be essential for a comprehensive investigation. Although many studies have investigated a variety of EBC candidate materials corroded by CMAS, such as rare-earth silicate and BSAS [13,17], no literature mentions mullite, which is the earliest and most widely used EBC. Therefore, in the present work, real VA was used for high-temperature corrosion with dense mullite ceramic bulk, which was fabricated by spark plasma sintering (SPS). This study aims to investigate the influence of VA, explain how VA attacks mullite ceramic, and predict the corrosion resistibility of mullite EBCs to VA.

2. Experimental procedures

2.1. Characterization of Icelandic VA and sintered mullite

In this study, the VA from the Icelandic volcano was selected. The VA was characterized well, because the chemical composition of VA varies geologically. Therefore, characterizing the basic properties, such as composition, phase, and melting temperature, of the Icelandic VA used in this study was necessary. The compositions, including the elements and oxide mass, were estimated by using inductively coupled plasma-optical emission spectroscopy (ICP-OES, Shimadzu ICPS-8100), and phase components and their crystallinity were checked by X-ray diffraction (XRD, Rigaku Smart Lab 9 kW, Cu $K\alpha_1$: 1.54056 Å). The melting point of Icelandic VA was measured by thermo gravimetric analysis and differential scanning calorimetry (TG-DSC, Linseis STA-PT1600).

To obtain highly dense sintered mullite, SPS equipment was utilized, and commercial mullite powder (KM 102, KCM Co., Japan) was placed in a graphite mold with a diameter of 30 mm. The mold was placed in a spark plasma apparatus (Syntex Inc., Japan), and the temperature was raised to 1400 °C in a vacuum with 10^{-2} Pa pressure. Sintering was conducted under 60 MPa of pressure. Afterwards, we used Archimedes method to measure the density of SPS fabricated mullite. Result shows that SPS fabricated mullite possesses approximately 97% densification compared with theoretical density. Phase analyses of the mullite powder and the SPS sintered body were characterized by XRD (Rigaku D/MAX-2200, Cu $K\alpha$: 1.5418 Å) in the 2θ range of 30–70° at a scan rate of 3°/min under air and at room temperature.

2.2. High-temperature corrosion behavior

The sintered mullite was cut with a diamond saw into 10 mm×5 mm specimens with a thickness of approximately 3 mm. After ultrasonic cleaning with acetone and ethanol, the VA was

Table 1

Chemical composition of Icelandic VA measured by ICP-OES. (Upper: elemental basis; bottom: oxide basis).

Element	Si	Al	Fe	Na	Ca	Mg	K	Ti	Mn	P
Mass%	27.1	7.9	6.8	3.9	3.8	2.1	1.7	0.9	0.2	0.1
Oxides	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	CaO	MgO	K_2O	TiO_2	MnO	P_2O_5
Mass%	58.1	14.9	9.7	5.3	5.3	3.4	2.1	1.5	0.2	0.3

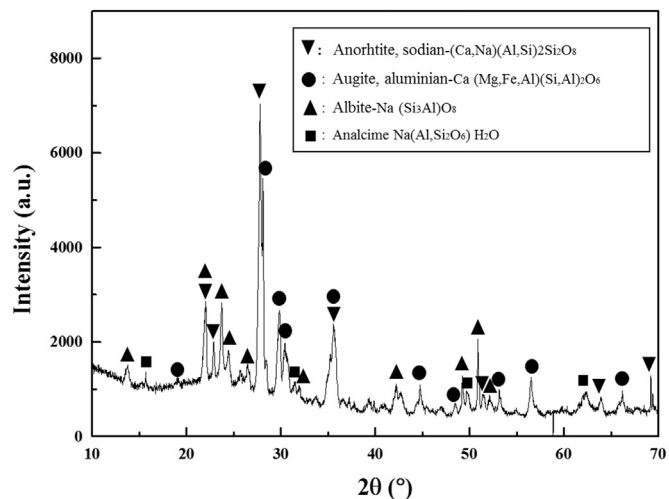


Fig. 1. XRD pattern of Icelandic volcanic ash.

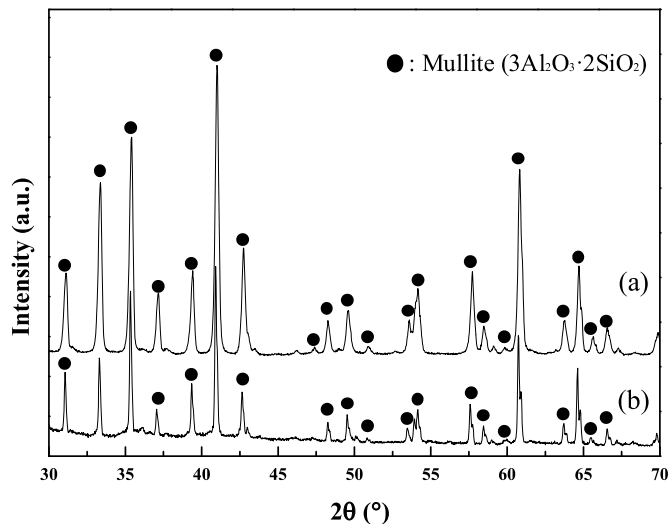


Fig. 2. XRD patterns of (a) mullite powder and (b) spark plasma sintered mullite.

attached to the polished surface of each cut mullite specimen by using a paste state with a concentration of 40 mg/cm². High-temperature corrosion testing with VA was carried out in a laboratory environment by using a box furnace. Duration times for the corrosive at 1400 °C were 2, 12, and 48 h; the heating rate was raised to 1400 °C at 5 °C/min. After high-temperature corrosion, the corroded mullite specimens were naturally cooled to room temperature. The corrosive phenomena and elemental analyses of each specimen were investigated by using a scanning electron microscope and energy dispersive X-ray spectrometry system (SEM-EDS, JEOL JSM-7001F) with an acceleration voltage of 20 kV.

The reaction temperature of VA and mullite was measured by TG-DSC. VA and mullite mixed powders were stirred evenly. The heating profile in this experiment was set from room temperature (around 25 °C) to 1400 °C at a heating rate of 10 °C per minute. The chamber

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