



Letter

Aluminum titanate-calcium dialuminate composites with low thermal expansion and high strength



A B S T R A C T

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Aluminum titanate (Al_2TiO_5 , AT) has many advantages, such as high melting point, low thermal expansion and low thermal conductivity, but its industrial applications are hindered by phase decomposition at high temperature and low mechanical strength. This paper reports the properties of calcium dialuminate (CaAl_2O_7 , CA_2) reinforced AT matrix composites. With the aid of Fe and Mg doping, the phase decomposition of AT is prevented. The three point bending strength of the composites is enhanced remarkably with increasing CA_2 content. After sintering at 1400 °C, the AT-40wt% CA_2 sample had a bending strength of 35 MPa about two orders of magnitude higher than that of the pure AT sample (~0.3 MPa). The well sintered AT- CA_2 composites also exhibit low thermal expansion coefficient, especially for the 1400 °C sintered AT-20wt% CA_2 sample ($0.23 \times 10^{-6}/^\circ\text{C}$). The AT- CA_2 composite with high mechanical strength and low thermal expansion is promising candidate in high temperature applications.

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1. Introduction

Aluminum titanate ceramics (Al_2TiO_5 , AT) with a crystal structure of pseudobrookite (Fe_2TiO_5) [1] has great interest for high temperature structural applications owing to high melting point of 1860 °C, low thermal expansion coefficient (TEC) of $\sim 1 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$, low thermal conductivity of 0.9–1.5 $\text{Wm}^{-1}\text{K}^{-1}$ and excellent thermal shock resistance [2–4]. Hence, it is desired to be used as diesel particulate filters, gasoline automobile exhaust catalyst carriers and diesel engine cylinders [5–8]. However, the industrial applications are hindered by two drawbacks. One is the phase decomposition into $\alpha\text{-Al}_2\text{O}_3$ and TiO_2 -rutile within the temperature range of 800–1280 °C [9]. The other is the poor fractural strength due to extensive microcracks [10,11]. Many works have been devoted to eliminate the drawbacks by doping additives [12–17]. MgO is one of the most popular additives to prevent the phase decomposition of AT by forming MgTi_2O_5 [12–15]. For example, Tsetsekou [12] studied the decomposition behavior of MgO stabilized AT, while Pratapa [13] researched the decomposition rate of $\text{Al}_2(1-x)\text{Mg}_x\text{Ti}_1+x\text{O}_5$. Fe_2O_3 is another efficient doping to prevent the decomposition of AT [16], where stable Fe_2TiO_5 is formed. In addition, many other additives were studied, such as Mn, Cr and Co [17]. However, the enhancement of fractural strength was always accompanied by the degradations of other properties, especially an obviously increment in TEC. This paper studied the preparation and properties of calcium dialuminate (CaAl_2O_7 , CA_2) reinforced AT matrix composites, where the co-doping of Mg and Fe was used to prevent the decomposition of

AT. The mechanical strength was greatly improved and TEC value was still low.

2. Experimental procedure

The AT and CA_2 were prepared by conventional solid–state reaction method. With the raw materials of $\alpha\text{-Al}_2\text{O}_3$ (>99%, median particle size 53 μm) and CaCO_3 (>99%), Fe_2O_3 and MgO stabilized AT was calcined at 1500 °C for 5 h. It was mixed with 10–40 wt% CA_2 and ground in planetary ball milling for 3 h. The resultant powders with 5 wt% binder of PVA was pressed uniaxially under a pressure of 3 MPa for disc samples (15 mm diameter) and rectangular samples (45 mm \times 10 mm \times 5 mm). After dried at 120 °C, the samples were sintered in the temperature range from 1250 °C to 1400 °C for 5 h.

The phase composition was characterized using an X-ray diffractometer (XRD, Rigaku DMAX-RB, Cu $K\alpha$ radiation) for the sintered disc samples. The microstructure of the fracture surfaces of sintered samples was observed using a scanning electron microscopy (SEM, LEICA S440i). The linear TEC was measured in a horizontal dilatometer within the temperature range of 25–1000 °C at a heating rate of 10 °C/min.

Room temperature flexural strength was measured by a three-point bending method in a universal materials testing machine (cdw-5). The measurement used bar-shaped specimens with dimensions of 40 mm \times 4 mm \times 3 mm and span length of 30 mm, while the crosshead speed was 0.5 mm min^{-1} . Before measurements all samples were polished and beveled by 400-

mesh to 1500-mesh metallographic sandpapers to eliminate surface stress.

3. Result and discussion

Fig. 1(a) shows the XRD spectra of AT-30wt%CA₂ samples sintered at different temperatures. After sintering at 1250 °C for 5 h, AT and CA₂ are formed by the solid state reaction there is still a lot of Al₂O₃ and TiO₂ residual amounts in the specimen. With the rise of sintering temperature, the residual amount of Al₂O₃ and TiO₂ reduce gradually. The specimen sintered at 1350 °C only contains AT and CA₂ phases, without any other phase. The peak intensity of CA₂ phase enhances with increasing sintering temperature. Fig. 1(b) shows the XRD spectra of the 1350 °C sintered samples with different CA₂ content. Same as the AT-30wt%CA₂ sample, the AT-10wt%CA₂ and AT-20wt%CA₂ samples have almost all AT and CA₂ phases, while the AT-40wt%CA₂ has tiny Al₂O₃ residual amounts. In addition, XRD result does not show obvious phase decomposition of AT even after 30 h dwelling at 1250 °C due to the aid of Fe and Mg.

Fig. 2 shows the measured density of AT-xwt%CA₂ samples sintered at different temperatures. The density increases with the rise of CA₂ amount or sintering temperature due to the grain growth and the boundary motion during the mullitization. With increasing CA₂ content, the density increases monotonically, indicating that the positive effect of CA₂ addition on densification.

Fig. 3(a–d) are SEM photos in secondary electron mode showing the microstructure of the surfaces of the sintered AT-CA₂ composites. The AT grains and CA₂ grains are distributed homogeneous in the samples. The AT and CA₂ phases can be distinguished by their shape and size, which is also confirm by elemental analysis. The AT phase has larger grains with an average length of ~20 μm (as indicated by arrows 1), while CA₂ grains are as small as about 3 μm (as indicated by arrows 2). The EDX analysis for point 1 confirms that large grain in the matrix is mainly composed of Al, Ti and O, i.e. AT, while that for point 2 confirms that small grains is mainly composed of Al, Ca and O, i.e. CA₂. The number of small CA₂ grains increases with the rise of CA₂ phase content. In addition, there are many microcracks in the AT matrix, which was produced during high temperature dwelling and cooling process. It is because TEC values of AT are remarkably different in various crystalline directions. The arrows 3 indicate intergranular microcracks, while the arrow 4 indicates a transgranular microcrack. There are also some pores distributed uniformly throughout the matrix, as indicated by white arrow 5. The existence of microcracks plays an important role in the low TEC of AT. A dense microstructure with less microcracks may work for higher mechanical strength, which always

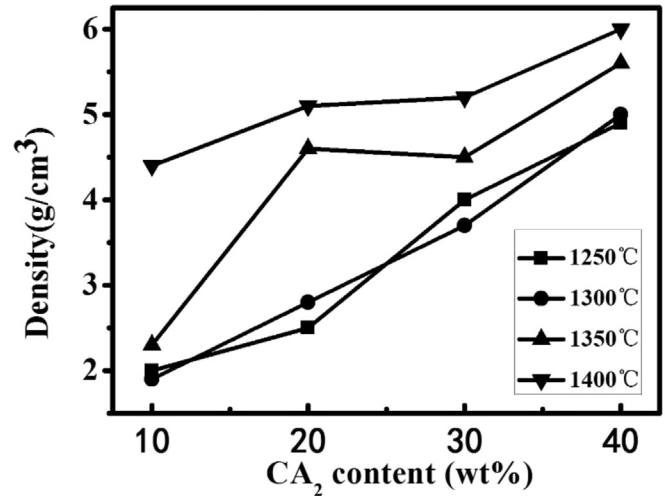


Fig. 2. Density of AT-xwt%CA₂ samples sintered at different temperatures.

induces a remarkable increase of TEC.

Fig. 4 shows the flexural strength of the samples with different CA₂ content and sintered at different temperatures. The strength of pure AT is 0.27 MPa, 0.31 MPa, 0.21 MPa and 0.26 MPa after sintering at 1250 °C, 1300 °C, 1350 °C and 1400 °C, respectively. The very low mechanical strength of AT ceramics is closely related to the existence of microcracks in the microstructure of the sintered samples. With the addition of CA₂, the flexural strength enhances obviously, especially for the samples sintered at higher temperatures. As CA₂ content increases from 0 to 40 wt% the flexural strength increases two orders of magnitude, from 0.26 MPa to 35 MPa. The strengthening mechanism involves the reinforce doping [18–20] into the matrix, its dispersion along AT's grain boundaries and the densification [21] of sintered samples. CA₂ is selected in this composite due to the positive thermal expansion coefficient, high flexural strength and high melting point, and it also acts as a crack stopper to alleviate the crack propagation. The density shows a similar variation trend as the flexural strength (Figs. 2 and 5). Less CA₂ content can lower densification obviously while high density is always accompanied by high flexural strength.

Fig. 5 represents the mean TEC values within a temperature range from 25 °C to 1000 °C for the samples with different CA₂ content and sintered at different temperatures. Thermal expansion curves of AT-CA₂ composites demonstrate a combined effect of the expanding behavior of CA₂ and the contracting behavior of AT. In general, the higher sintering temperature or the more CA₂

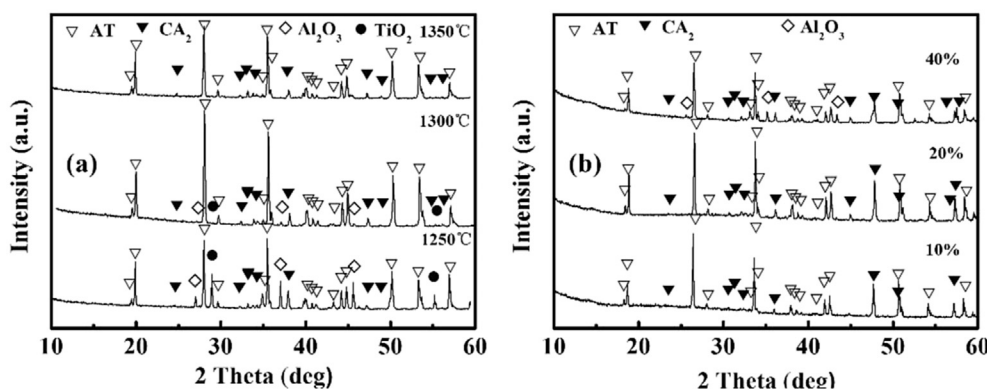


Fig. 1. XRD spectra of (a) the AT-30wt%CA₂ samples sintered at different temperatures and (b) the AT-x wt% CA₂ samples sintered at 1350 °C.

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