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Interface evolution of the C_f/leucite composites derived from C_f/geopolymer composites

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Abstract

In this paper, effect of heat treatment temperature on the interface structure of the carbon fiber reinforced geopolymer composites was investigated by transmission electron microscopy (TEM), selected-area diffraction (SAD) analysis and high-resolution transmission electron microscopy (HRTEM). In the composite treated at 1100 °C, carbon fiber showed a good bond with the leucite matrix and no interface reaction layer was observed, while due to the thermal mismatch between fiber and matrix, microcrack which was perpendicular to the fiber axial direction can be seen in the matrix. With increase in heat treatment temperature to 1200, 1300 and 1400 °C, interface reaction occurred and reaction layers with thickness of 50, 100 and 1000 nm, respectively, were detected. The interface layer was formed by the reaction between Si–O groups in the matrix and C element in the fiber. Especially for the sample treated at 1400 °C, serious interface reaction led to the formation of β-SiC and property of carbon fiber was greatly degraded.

Keywords: B. Interface; Carbon fiber; Leucite; Geopolymer

1. Introduction

Geopolymer materials have been developed for many years and many applications have been found for their excellent properties, like fire resistance, low density, low cost, easy processing, environmentally friendly nature and thermal resistance [1–5], and low strength and brittle fracture nature are the most serious impedances which limit their use in structural applications.

Over the past years, various kinds of reinforcements, including particulate [3,4], continuous fiber [5–9] and short fiber [10,11] reinforced geopolymer composites have been extensively investigated. Among them, continuous fiber reinforced geopolymer composites have generated a great deal of attention due to their adaptability to conventional polymer composites manufacturing techniques. Meanwhile, the high strength and modulus of the fibers can prevent catastrophic brittle failure in composites. However, due to the very weak geopolymer matrix, fiber reinforced geopolymer composites still showed low mechanical properties [3,12,13], which limit their wide applications.

Recently, many sources proved that geopolymer can be converted into advanced ceramics with high strength and stiffness by heat treatment [14–22]. In our recent research [23] we reported that after proper high temperature heat treatment, continuous carbon fiber reinforced geopolymer composite can be directly converted into carbon fiber reinforced leucite composite and mechanical properties can be greatly enhanced. The composite being treated at 1100 °C showed the highest flexural strength of 234.2 MPa, which was caused by the densified and crystallized matrix and the enhanced fiber/matrix interface bonding based on the fine-integrity of carbon fibers, and with increase in heat treatment temperature to 1200, 1300 and 1400 °C, flexural strength decreased to be 181.7, 160.3 and 54.6 MPa, respectively. Heat treatment temperature has a great effect on the interface state between leucite and carbon fiber, which played an important role in determining the whole

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performance of the composite. Thus, in this paper, interface structure and composition evolution caused by physical and chemical compatibility between fiber and matrix are further investigated by the TEM technique, which provides further scientific proof to support the evolution of mechanical properties of the composite with increasing treatment temperature.

2. Experimental procedures

Geopolymer resin was obtained by mixing metakaolin powder with a potassium silicate solution, as described in our previous publication [23]. The carbon fiber used in this study (Jilin Carbon Indus., China) is unidirectional continuous carbon fiber and has a diameter of 6–8 μm, and tensile strength of 2930 MPa. The composite was prepared by infiltrating geopolymer resin into the unidirectional continuous PAN-based carbon fiber preform with the help of the ultrasonic vibration treatment, and stacked one by one to get a green sample with 16 layers. To remove the pores in the green compact, degassing was applied at 80 °C for 24 h using a vacuum-bag technique. After that, the composite sample was cut into 4 parts, and each was placed at 1100, 1200, 1300 and 1400 °C, respectively, for 90 min in an argon atmosphere, to get the carbon fiber reinforced leucite composites. Composites treated at 1100, 1200, 1300 and 1400 were denoted as C-1100, C-1200, C-1300 and C-1400, respectively.

The interface microstructures were characterized by transmission electron microscopy (TEM), selected-area diffraction analysis and high-resolution transmission electron microscopy (HRTEM). Thin foil specimens taken parallel to the fiber axis were prepared by dimpling and subsequent ion-beam thinning.

3. Results and discussion

Fig. 1 shows the TEM micrographs and SAD results of C-1100. It could be observed that C_f had a good bond with the leucite matrix without obvious interfacial reaction or amorphous layer, indicating no chemical reaction between fiber and matrix occurred during composite processing. SAD result as shown in Fig. 1(b) indicated the carbon fiber was amorphous, and SAD from matrix displayed in Fig. 1(c) confirmed the formation of the crystallized tetragonal leucite, which was consistent with the XRD results in our previous publication [23].

However, microcrack in the matrix which was perpendicular to the fiber axial direction can be observed. The microcrack was mainly derived from the thermal mismatch between carbon fiber and leucite matrix during the cooling process. The average coefficient of thermal expansion (CTE) of the leucite matrix is $14.74\times10^{-6}\,^{\circ}\mathrm{C}^{-1}$ from 30 °C to 1300 °C, which is higher than those of carbon fiber in both radial direction ($10\times10^{-6}\,^{\circ}\mathrm{C}^{-1}$) and axial direction ($0\times10^{-6}\,^{\circ}\mathrm{C}^{-1}$). Thermal mismatch would lead to the residual stress in the composite in both radial and

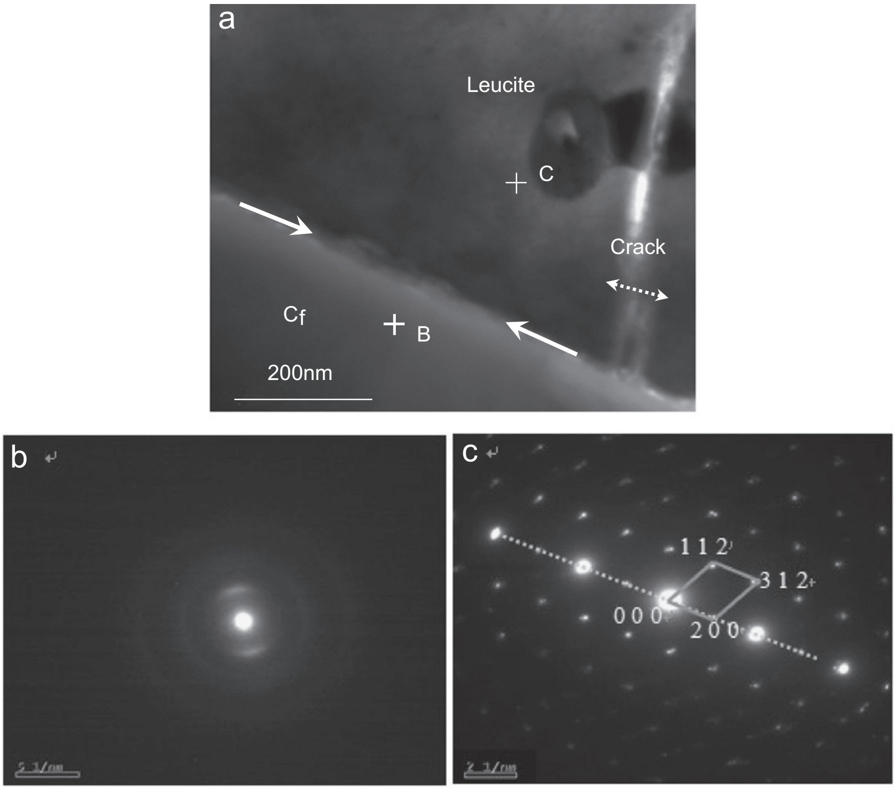

Fig. 1. Interface morphology of C-1100: (a) TEM image, (b) and (c) microdiffraction analyses of carbon fiber and matrix in area of B and C.