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Ceramics International 36 (2010) 1447–1453
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Effects of high-temperature heat treatment on the mechanical properties of unidirectional carbon fiber reinforced geopolymer composites

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Received 19 November 2009; received in revised form 5 December 2009; accepted 26 January 2010
Available online 1 March 2010

Abstract

Unidirectional carbon fiber reinforced geopolymer composite (C_{uf}/geopolymer) is prepared by a simple ultrasonic-assisted slurry infiltration method, and then heat treated at elevated temperatures. Effects of high-temperature heat treatment on the microstructure and mechanical properties of the composites are studied. Mechanical properties and fracture behavior are correlated with their microstructure evolution including fiber/matrix interface change. When the composites are heat treated in a temperature range from 1100 to 1300 °C, it is found that mechanical properties can be greatly improved. For the composite heat treated at 1100 °C, flexural strength, work of fracture and Young's modulus reach their highest values increasing by 76%, 15% and 75%, respectively, relative to their original state before heat treatment. The property improvement can be attributed to the densified and crystallized matrix, and the enhanced fiber/matrix interface bonding based on the fine-integrity of carbon fibers. In contrast, for composite heat treated at 1400 °C, the mechanical properties lower substantially and it tends to fracture in a very brittle manner owing to the seriously degraded carbon fibers together with matrix melting and crystal phases dissolve.

Keywords: B. Composite; C. Mechanical properties; Geopolymer; Unidirectional carbon fiber

1. Introduction

Geopolymer materials have attracted a lot of attention for various applications due to their excellent fire resistance, low density, low cost, easy processing, environmentally friendly nature and excellent thermal properties [1–5]. Compared with polymer, geopolymer materials can be used under much more higher temperature like 1000 °C and they tend to be uncombustible and no poisonous smoke would be released [6]. Recently geopolymer materials have shown significant promise as aircraft cabin and heat resistant materials, and have been investigated as an alternative to polymer composites [6,7].

On the other hand, like ceramics, the pure geopolymer matrix exhibits relatively low mechanical properties, and hence limits its use in structural applications. Over the past years, various kinds of geopolymer based composites, including particulate [3,4], continuous fiber [5–9] and short fiber [10,11] reinforced geopolymer composites have been extensively investigated. 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 of Si/Al ≤ 2 composites still showed low mechanical properties [3,12,13], which is a great impediment to their wide applications.

Recently, some people concentrate their attention on the formation of ceramics from geopolymer of Si/Al ≤ 2 [4,14–19]. After being heat treated geopolymer will convert into crystalline phases which have excellent mechanical and thermal properties [15,16]. Based on the densification and crystallization of geopolymer upon heating, mechanical properties of the composites should be further improved.

According to literature, the effect of heat treatment on mechanical properties of continuous carbon fiber reinforced geopolymer matrix composite has not yet been reported so far. In the present work, therefore, C_{uf}/geopolymer composites were prepared by a simple ultrasonic-assisted slurry infiltration method, and then treated at 1000, 1100, 1200, 1300 and

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0272-8842/$36.00
doi:10.1016/j.ceramint.2010.02.012

Table 1
Chemical composition of metakaolin (wt%).

Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Fe_2O_3	Σ
42.53	54.64	0.16	0.11	0.48	0.12	0.80	0.97	99.81

1400 °C, respectively. The effects of heat treatment temperature on the evolution of phase composition, microstructure and fiber/matrix (C_f/matrix) interface condition are studied. Mechanical properties and fracture behavior are correlated with microstructure evolution including C_f/matrix interface.

2. Experimental procedures

Geopolymer resin with composition of $SiO_2/Al_2O_3 = 3$, $K_2O/SiO_2 = 0.33$ and $H_2O/K_2O = 11$ (mole ratio) was obtained by mixing metakaolin powder with a potassium silicate solution. Chemical compositions of the metakaolin (800 °C calcined kaolin, Shanghai Fengxian Indus., China) were given in Table 1. The main phase of metakaolin was amorphous with a characteristic hump centered at about 23° in 2θ and its minor phase was α-quartz. The potassium silicate solution was prepared by dissolving amorphous silica (Shanghai Dixiang Indus, China) into a KOH (Tianjin Fuchen Indus., China) solution. The solution was then allowed to mature under stirring for 48 h in order to dissolve the silica completely [12].

The carbon fiber used in this study (Jilin Carbon Indus., China) has a diameter of 6–8 μm, and its properties are summarized in Table 2. 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 5 parts, 4 of which were placed at 1000, 1100, 1200, 1300 and 1400 °C, respectively, for 90 min in an argon atmosphere. Composites without and after heat treatment were denoted as C-W, C-1000, C-1100, C-1200, C-1300 and C-1400, respectively. Meanwhile, geopolymer matrix was also heat treated at 1100 °C to obtain leucite ceramic. The content of carbon fibers was about 20 vol% for composite without heat treatment, and 25 vol% for composites after heat treatment due to the densification of the matrix.

Phase compositions of the samples were examined by X-ray diffractometer (XRD, Rigaku, RINT–2000). Thermogravimetric analysis (TGA, Netzsh STA 409 instrument) were carried out in an atmosphere of flowing argon in alumina crucibles over a temperature range from 35 to 1400 °C. Flexural strength and Young's modulus measurements of composites and geopolymer matrix and leucite ceramics were conducted on specimens (4 mm × 3 mm × 36 mm) using a three-point-bending fixture on an Instron-500 tester with a span length of 30 mm at a crosshead speed of 0.5 mm/min. Specimens were machined with the tensile surface perpendicular to the direction of lamination. Load-displacement curves were recorded. Work of fracture was calculated by the area between the load curve and *X*-axis in the load/displacement curves till the load drops to 90% of the maximum load. Six specimens were tested under each test condition. Microstructure on the polished surface and fractograph of the composites were observed by scanning electron microscopy (SEM, FEI, Quanta-200).

Table 2
Typical properties of carbon fiber.

Diameter (μm)	Density (g cm^{-3})	Tensile strength (MPa)	Tensile modulus (GPa)
6–8	≥1.76	2930	200–220

3. Results and discussion

3.1. Phase characterization

XRD patterns for composites heated to a variety of temperature are shown in Fig. 1. C-W displayed typical amorphous XRD characteristic. In addition, several sharp characteristic peaks were also observed, which were identified as α-quartz introduced by metakaolin. For C-1000 sample, the XRD patterns remained predominately amorphous, indicating the non-crystallization of matrix. After composite was heat treated at 1100 °C, leucite ($K_2O{\cdot}Al_2O_3{\cdot}4SiO_2$) and a little kalsilite ($K_2O{\cdot}Al_2O_3{\cdot}2SiO_2$) appeared. For the samples being heat treated from 1100 to 1300 °C, leucite became a major phase. When heat treatment temperature was further increased to 1400 °C, a broad amorphous hump between 20° and 30° of 2-theta appeared which revealed the partial melting of matrix. Simultaneously the peak intensity of leucite decreased indicating the crystal phases dissolve. At 1400 °C it was also revealed that a little amount of β-SiC phase appeared in the composites, indicating that carbon fiber reacted with Si–O unites into SiC phase at an enough high temperature.

Fig. 2 shows the TG curves of the geopolymer and obtained C_{uf}/geopolymer composite, which further confirms that interface

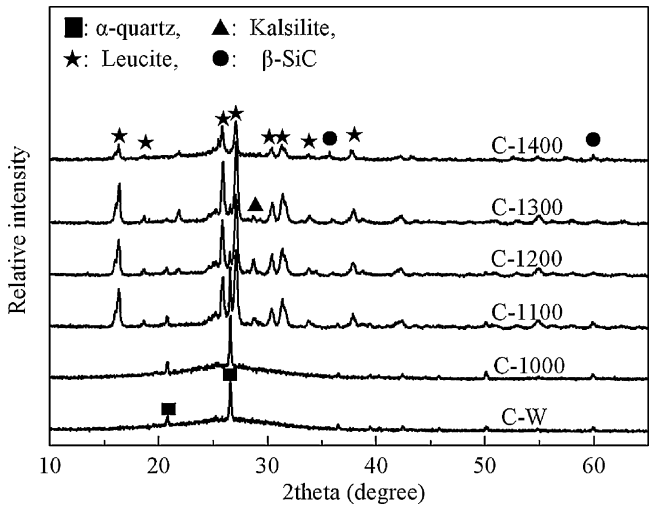

Fig. 1. XRD patterns of C_{uf}/geopolymer composites without and after heat treatment.

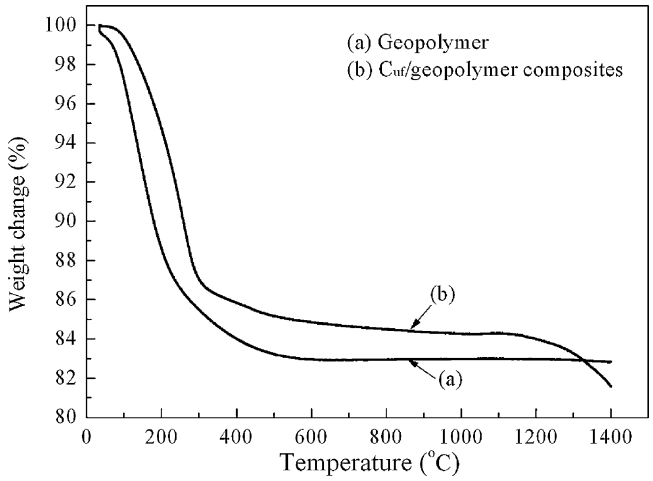

Fig. 2. TGA curves of geopolymer and C_{uf}/geopolymer composites.

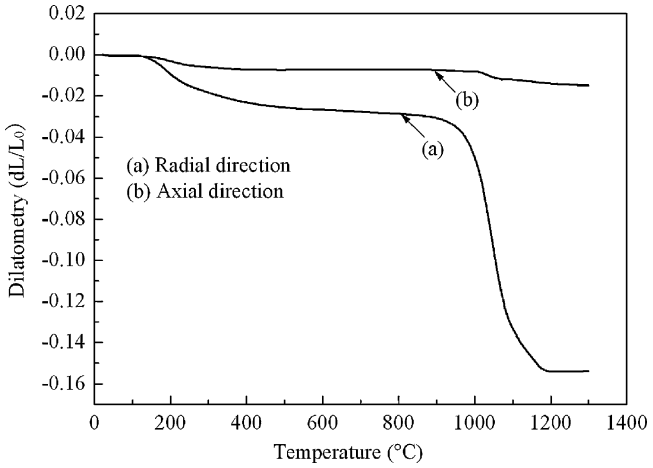

Fig. 4. Thermal shrinkage of the composite in radial and axial direction.

reaction occurred at high temperature. The major weight loss before 700 °C from geopolymer and composite was resulted from water loss, by evaporation of both free water and condensed hydroxyl groups [4,15–18]. From 700 to 1400 °C, geopolymer showed little weight change. In contrast, for the composite there was another fast weight loss stage above 1170 °C which might be due to the interface reaction between carbon fiber and matrix. Considering the XRD analysis results, we can deduce that interface reaction was as given in formula (1) [20]:

$$Si{-}O + 2C \rightarrow SiC + CO_{(g)} \quad (1)$$

Interface reaction in the C_{uf}/geopolymer composite most likely started at 1170 °C. From 1170 to 1400 °C, the weight loss was about 3 wt%, suggesting that carbon fibers might have been seriously degraded.

3.2. Microstructure characterization

Fig. 3 shows typical microstructures of composites after heat treatment. After the composite was heat treated at 1100 °C, many short oval cracks in radial direction of carbon fiber were formed. These oval cracks were resulted from the large volume

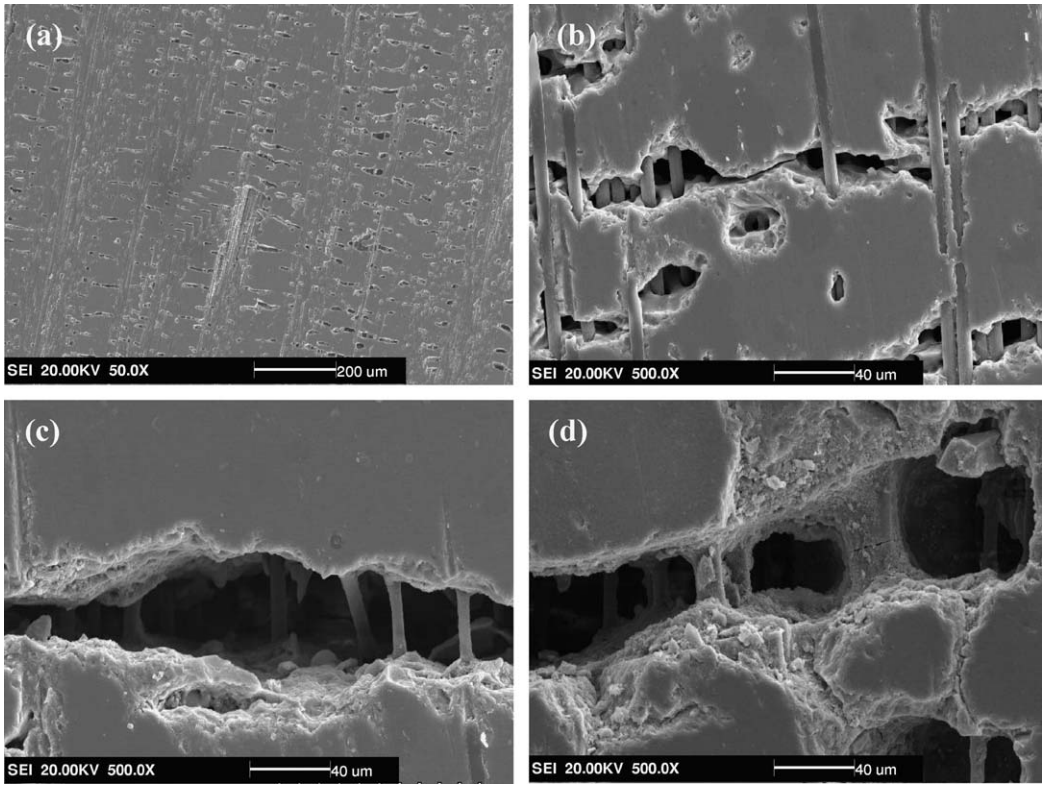

Fig. 3. Typical microstructure of polished cross-section of composites: (a) C-1100 (low magnification); (b) C-1100 (high magnification); (c) C-1300 and (d) C-1400.

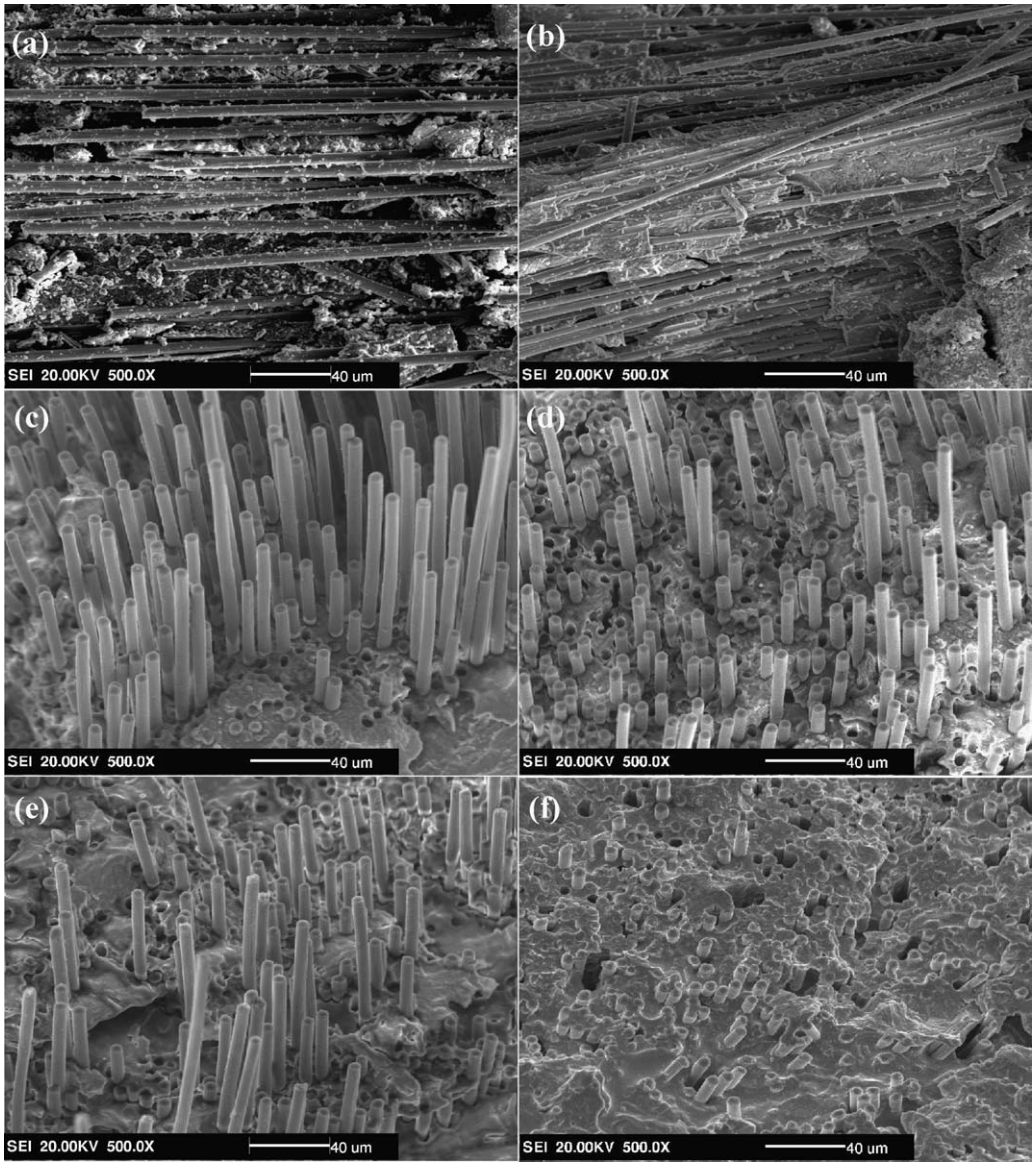

Fig. 5. Typical microstructure of fracture surface of C_{uf}/geopolymer composites without and after heat treatment: (a) C-W; (b) C-1000; (c) C-1100; (d) C-1200; (e) C-1300 and (f) C-1400.

shrinkage caused by viscous sintering of geopolymer matrix at high temperature [4,15–18]. As shown in Fig. 4, on heating from 900 to 1200 °C the thermal shrinkages (dL/L_0) in radial and axial direction are 12.4% and 0.9%, respectively. Matrix shrinkage in axial direction was highly hindered by the carbon fibers, thus led to the formation of the evenly distributed oval cracks in the softened matrix. For C-1100 sample, Fig. 3 (b) indicated that the cracks were fully bridged by fibers which maintained fine-integrity. With increasing heat treatment temperature, the thermal mismatch between fiber and matrix increased and the interfacial reaction aggravated, so the thermally mismatched cracks opened more wide and fibers were damaged more seriously as shown in Fig. 3(c) and (d).

These cracks would undoubtedly decrease the mechanical properties and oxidation resistance of composites. Therefore, it was necessary to find an effective way to eliminate them. Incorporation of cesium into leucite and repeated infiltration of matrix slurry into composites were two efficient methods. The incorporation of cesium would significantly lower the thermal shrinkage of geopolymer [21–23] and thus enhance the composites, and repeated infiltration could have a healing effect on the cracks thus decrease their number and size [24–27], which would be discussed in a later research.

3.3. C_f/matrix interface evolution

C_f/matrix interfacial structure is an important factor to determine mechanical properties of composites [28–30]. And it can be evaluated by the morphology of fracture surfaces [27], as shown in Fig. 5. For the samples C-W and C-1000 (Fig. 5(a) and (b)), most fibers did not fracture and fragmentation of geopolymer matrix distributed in the intra-bundles of fibers. Fiber debonding could also be observed, indicating the weak C_f/matrix interface. After composites were heat treated at

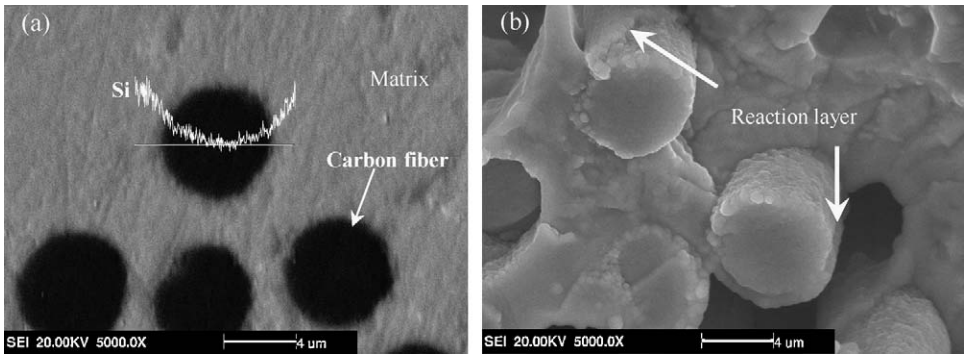

Fig. 6. Characteristic X–ray line profiles of C and Si atom and interface reaction in sample C-1400.

Table 3
Mechanical properties of geopolymer and C_{uf}/geopolymer composites.

Samples	Flexural strength (MPa)	Work of fracture (J m^{-2})	Elastic modulus (GPa)
Matrix			
Geopolymer	12.3 ± 1.2	–	10.3 ± 1.2
Leucite ceramic	70 ± 6.8	–	65 ± 6.3
C-W	132.9 ± 8.2	3874.5 ± 266.8	36.5 ± 3.4
C-1000	95.6 ± 10.5	2354.8 ± 243.6	30.4 ± 4.1
C-1100	234.2 ± 22.6	4445.7 ± 263.1	63.8 ± 3.9
C-1200	181.7 ± 16.3	4233.4 ± 299.5	55.9 ± 4.8
C-1300	160.3 ± 19.2	2444.6 ± 283.2	50.6 ± 3.6
C-1400	54.6 ± 3.7	366.6 ± 37.5	41.5 ± 4.2

higher temperature, for the great shrinkage of matrix, frictional restraint stress at C_f/matrix interface would increase and interface bonding was enhanced. Fig. 5(c) shows that long fiber pull-out dominated the fracture surface and the fibers all maintained fine-integrity. For C-1200 and C-1300, fiber pull-out was still very clear on the fracture surface, though most of the pulled out fibers appeared to be a little shorter than that of the C-1100 sample. However, for the sample C-1400, the fracture surface was much more flat and few fibers pull-out could be observed. Extensive fiber pull-out of composites usually indicated a relatively weak C_f/matrix interfacial bonding, while little fiber pull-out and short pull-out length indicated a strong C_f/matrix interfacial bonding [28,31]. Therefore, the C_f/matrix interfacial bonding of C-1100 was more desirable than those in other samples and great reinforcement could be achieved. For the C-1400, this was not the case. To get further information, element distribution was analyzed and higher magnification SEM fractograph was recorded in Fig. 6. As shown in Fig. 6(a), the diffusion of Si atoms into carbon fiber takes place in the C_f/matrix interface zone. As indicated in Fig. 6(b), an interface reaction layer of about 1 μm in thickness was clearly observed. According to the aforementioned XRD analysis results in Fig. 1, it can be concluded the reaction layer should be β-SiC. Due to formation of the thick interfacial SiC layer, too much strong C_f/matrix bonding was formed and the carbon fiber were seriously degraded, as a result, the reinforcement effect from the carbon fibers must be decompensated.

3.4. Mechanical properties and fracture behavior

Table 3 and Fig. 7 present the mechanical properties of geopolymer, leucite ceramic (derived from geopolymer after heat treatment at 1100 °C, polycrystalline material) and composites. It was found that the leucite formation effectively strengthened the composite matrix from the starting 12.3 to 70 MPa. The effects of heat treatment on mechanical properties of composites were quite obvious. As compared with the C-W sample, after being treated at 1000 °C, mechanical properties of composite (C-1000) decreased by 30%. While after composite was heat treated at 1100 °C, flexural strength, work of fracture

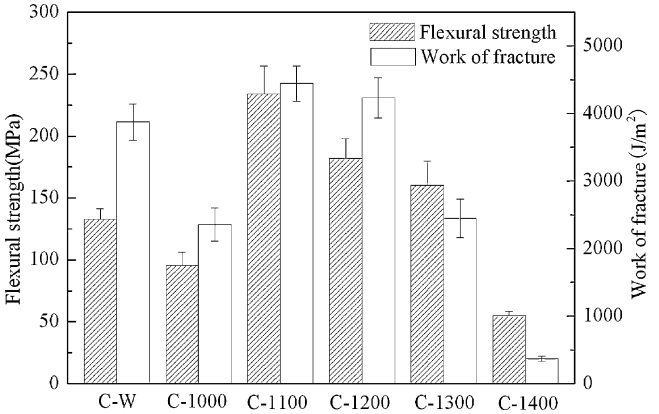

Fig. 7. Variations of flexural strength and work of fracture of C_{uf}/geopolymer composites without and after heat treatment at different temperatures.

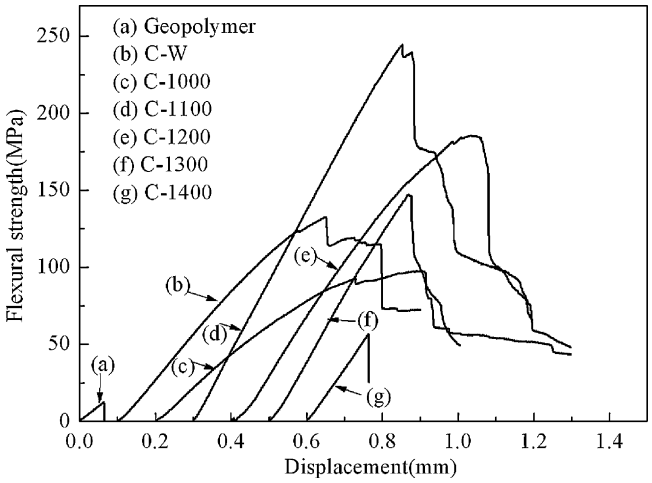

Fig. 8. Typical load-displacemen curves of geopolymer and C_{uf}/geopolymer composites without and after heat treatment.

and Young's modulus attained to their peak values, 234.2 MPa, 4445.7 J m^{-2} and 63.8 GPa, increasing by 76%, 15% and 75%, respectively, compared with that of C-W. With increasing heat treatment temperature from 1100 to 1300 °C, mechanical properties tended to decrease gradually but flexural strength and Young's modulus were still much higher than that of C-W and retained approximately 70–80% of the peak strength and 80–90% of the peak modulus. However, when heat treatment temperature was further increased to 1400 °C, flexural strength, work of fracture and Young's modulus of composite decreased sharply to 54.6 MPa, 366.6 J m^{-2} and 41.5 GPa, respectively, and it was in well agreement with the aforementioned effect of the SiC interface reaction layer on the mechanical properties. It should be mentioned that after heat treatment the fiber volume fraction for the composites increased relatively from the starting 20 to 25 vol% due to the shrinkage of the matrix. According to composite mixing rules, the calculated strength should be around 164.5 MPa which was still far lower than that of C-1100 sample. So, the increased fiber fraction had only a minor effect on mechanical properties of the composites.

Fig. 8 shows typical curves for flexural strength versus displacement of the geopolymer and C_{uf}/geopolymer composites before and after heat treatment. Geopolymer indicated a typical brittle failure mode (Fig. 8(a)). For the composites except C-1400 (Fig. 8(g)), no catastrophic failure was observed and they all exhibited elastic region and non-linear region. For C-1100, C-1200 and C-1300 (Fig. 8(d)–(f)) there were several significant steep drop-steps after reaching the maximum indicating the composites fractured in tensile fracture mode. While the strength–displacement curves of C-W and C-1000 (Fig. 8(b) and (c)) were different from the above three. The strength tended to reduce slowly after the maximum load, and then, the strength–displacement curves spread more widely indicating the shear fracture mode rather than tensile fracture mode. In the case of C-1400, the fracture behavior was similar to that of the geopolymer, showing a catastrophic fracture behavior. These were in good consistent with the fractograph observations in Fig. 5.

C-W and C-1000 samples were typical weak matrix composites. Their weak mechanical properties could be attributed mainly to the low strength and stiffness of geopolymer matrix that had not crystallized, and the weak C_f/matrix interface bonding strength also played a negative role. For composites heated between 1100 and 1400 °C, a stronger composites matrix developed for leucite formation. Because there were many matrix cracks as shown in Fig. 3, the mechanical properties of composite after heat treatment were dominated strongly by the properties of the fibers and fiber matrix interface condition. For C-1100, fibers maintained fine-integrity and C_f/matrix interface bonding was just in a good state. When the matrix cracks propagated and encountered the carbon fibers, they were largely deflected, which led to debonding of the C_f/matrix interface and fiber pulled out from the matrix. All these factors eventually resulted in the highest mechanical properties and non-catastrophic fracture behavior of the composite. When heat treatment temperature was further increased, interface reaction between carbon fiber and Si–O unites started at 1170 °C according to XRD and TGA analysis. On the one hand, the reaction would degrade the mechanical properties of carbon fiber. This was primarily responsible for the decreased mechanical properties of C-1200, C-1300 and C-1400 samples. On the other hand, interface reaction would lead to a strong interface bonding between fiber and matrix. In the light of interface theory of ceramic matrix composites [32], strong C_f/matrix interfacial bonding tended to lead to the matrix microcracks directly cutting through the fiber with the increase of load. So, with increasing heat treatment temperature the fiber pull-out length became shorter and fewer fibers were pulled out. Especially for C-1400, strengthening effect of fiber was dramatically lowered and catastrophic failure behavior was observed. Meanwhile, matrix melting and crystal phases dissolve also had detrimental effects on the mechanical properties. Thus, in composites after high-temperature heat treatment, C-1400 sample showed the lowest flexural strength, Young's modulus and work of fracture.

4. Conclusions

(1) Mechanical properties of the unidirectional carbon fiber reinforced geopolymer composite can be greatly improved by heat treatment in a wide range of temperature from 1100 to 1300 °C. Especially for C-1100, flexural strength, work of fracture and Young's modulus get to their peak values, 234.2 MPa, 4445.7 J m^{-2} and 63.8 GPa, respectively, and the composite fractures in a very gentle way. The increase in mechanical properties could be contributed to the matrix densification, and leucite formation as well as the proper C_f/matrix interface bonding strength.

(2) When the composite are heat treated at 1400 °C, the strengthening effect of carbon fiber is dramatically decompensated. It is resulted from the serious fiber degradation and the much strong fiber/matrix interface bonding strength. So, the composite shows substantially decreased mechanical properties and fractures in a very brittle manner.

Acknowledgements

This work was supported by Program for NewCentury Excellent Talents in University (NCET, Grant No. NCET-04-0327), Program of Excellent Team in Harbin Institute of Technology and the Science Fund for Distinguished Young Scholars of Heilongjiang Province.

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