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ADVANCES IN GEOPOLYMER SCIENCE & TECHNOLOGY

Novel geopolymer based composites with enhanced ductility

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Abstract Geopolymer materials have been proposed for various applications due to their excellent fire resistance and low processing cost. One requirement that geopolymer composites need to meet for structural applications is graceful failure, as catastrophic failure during service can result in significant loss of life. In this paper, the properties of novel low cost composites made by infiltrating stainless steel mesh with geopolymer resin are reported. Four point flexure tests on these composites showed metal-like yielding behavior instead of catastrophic failure and the ‘‘yield strength’’ was significantly higher than the flexure strength of the geopolymer matrix. Exposure to high temperatures, 800 and 1,050°C, resulted in the ‘‘yield strength’’ decreasing to ~59 and ~44% level respectively compared to the strength of as-processed samples. However, the good ductility was still retained after the high temperature treatment, which makes this novel composite a strong candidate for the applications where catastrophic failure upon fire/high temperature exposure needs to be avoided.

Introduction

Geopolymer materials have attracted a lot of attention for various applications due to their excellent fire resistance, low curing/hardening temperatures, low air and water permeability, and environmental durability [1]. One requirement that geopolymer composites need to meet for construction applications is graceful failure, as catastrophic failure during service can result in significant loss of life. In addition, if graceful failure could be achieved in geopolymer composites, this would open the window to a variety of applications where metals or polymer composites are used such as pressure vessel liners. Different types of laminates and composites made with geopolymer matrix materials and several different kinds of fibers had been developed for the applications where the fire resistance is a big concern [2–5].

The objective of this paper is to combine the high strength and stiffness of metal wires with the high temperature and chemical stability of geopolymers to develop a novel low cost composite. In the past, Lankard and Mobasher et al. had respectively shown that the toughness and strength of Portland cement concrete were significantly improved by reinforcing the concrete with either discontinuous steel fibers [6] or uniaxial, continuous polypropylene fibers [7]. So it is expected to see this reinforcement effect in the geopolymer system too. Giancaspro et al. [8] had fabricated composite laminates from geopolymer and metal mesh, while they used coarse metal mesh (20 or 40 mesh) combined with glass fibers and only room temperature properties were tested. Fine (120 mesh) stainless steel meshes were used in this paper to investigate the reinforcement effect of the ductile material. More importantly, the flexure

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behaviors of the composites after high temperature exposure were also reported to explore the potential for applications under possible high temperature/fire exposure conditions.

Experimental

Materials

Plain woven stainless steel mesh (mesh count 120, wire diameter 60 μm) was procured from Dorstener Wire Tech. Silica, alumina, and potassium silicate (K_2O:SiO_2 = 2.5:1 by weight) powders were procured from Alfa Aesar. All chemicals were used without further purification.

Composites processing

The geopolymer resin used here has a similar composition to what is described in the open literature [5], with Si/Al = 32:1 and K/Al = 7:1 (mole ratio). A typical processing route for the geopolymer resin is as follows: 18.32 g potassium silicate was mixed with 12.17 g distilled water by paint shaking for 10 min. Then 18.24 g silica (quartz) and alumina (corundum) powder was added and mixed in a paint-shaker again for another 10 min. For monolithic geopolymer samples, the slip was then cast in molds and dried at room temperature to form bars, and then cured at 80°C for 2 days.

To prepare composite samples, six stainless steel mesh layers (9 mm × 50 mm) were impregnated with the geopolymer resin and laid on top of each other. The lamination structure was formed by vacuum-bag technique and pressed at ~3 MPa for 1 h, followed by curing overnight at room-temperature and for an additional 2-days at 80°C. In some cases, the geopolymer resin was mixed with 10 vol% of Nextel 610 chopped alumina fibers before casting into bars or making composites in order to further strengthen the resin materials.

Measurement of flexure strength

The flexure strength of the bars was tested in four-point flexure using 20/40 mm inner and outer spans. The tests were conducted on an Instron system under extension control with an extension rate of 0.5 mm/min. Four or five samples for each type of specimens were tested to assure that reported results were typical and reproducible. However, only one stress–strain curve for each type of specimens is shown in the following figures for a better view.

Crystalline phase identification

The crystalline phases of the samples were identified by a Philips X'Pert X-ray diffraction system using Cu Kα radiation (1.5416 Å). A typical scan is from 10 to 80° with a scan rate of 0.02 deg/s.

Characterization of microstructure by SEM

The microstructures of the samples were observed by a JEOL JSM-5900LV SEM equipped with the EDS system for composition identification. The samples were sputtered with Au before SEM observations.

Characterization of microstructure by optical microscopy

The optical microscopy was conducted using a Nikon SMZ1000 optical system equipped with a Nikon DN100 digital camera to examine the cracks generated in the geopolymer matrix during testing.

Results and discussion

Flexure behavior of processed composites

Figure 1 shows the geopolymer–stainless steel mesh composite samples cured at 80°C before and after the flexure testing. The tested samples had significant

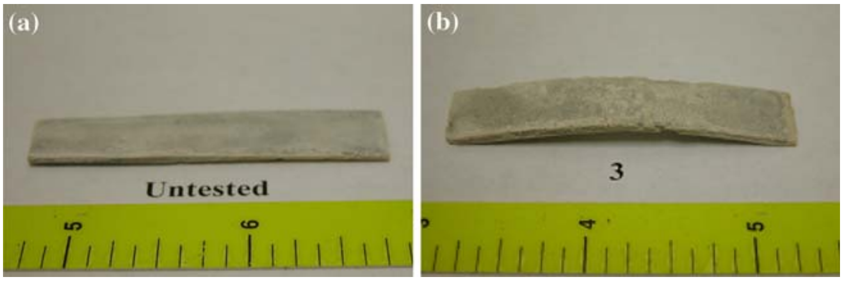


Fig. 1 Geopolymer–stainless steel mesh composite sample before (**a**) and after (**b**) a four-point flexure test

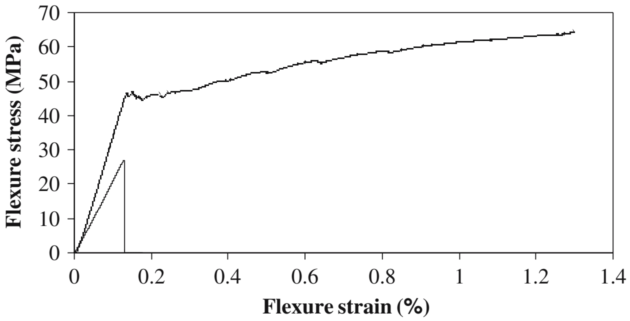


Fig. 2 Flexural stress–strain curves of geopolymer resin and geopolymer–stainless steel mesh composites cured at 80°C

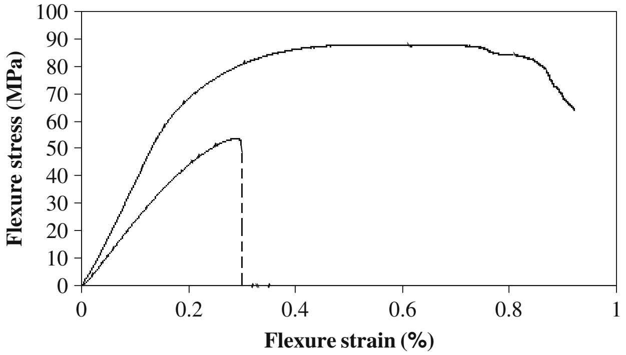


Fig. 4 Flexural stress–strain curves of chopped alumina fiber reinforced geopolymer resin and geopolymer–stainless steel mesh composite cured at 80°C

deformation but did not break or de-laminate. This superior ductility of the composite structure can also be seen from the flexure stress–strain curve (Fig. 2). As shown in Fig. 2, the geopolymer resin itself showed typically brittle behavior of ceramics and fractured at a flexure strain of 0.1% and a flexure strength of about 25 ± 2 MPa. However, the steel mesh reinforced geopolymer composite showed metal-like ductile behavior and the sample yielded at the stress level of 41 ± 5 MPa instead of fast fracture typical of ceramics. After yielding, the sample continued to deform but the stress only increased slowly. None of composite samples fractured even at over 1% of flexure strain where the testing had to be stopped due to the limitation of the testing fixture. Therefore, the composite is a much stronger and more ductile material compared to pure geopolymer resin. Figure 3 shows the optical images of the tensile and compression surface of tested samples. On the tensile side, cracks were generated with homogeneous spacing, but there was no apparent peel-off of the geopolymer resin indicating good bonding between the steel mesh and the matrix material. On the compression side, there were no macro-level cracks in spite of the large deformation.

Adding chopped alumina fiber, Nextel 610, into the geopolymer resin can further strengthen the matrix material, which is shown in Fig. 4. About 10 vol% of chopped Nextel 610 fiber increased the flexure strength of the resin from 25 ± 2 MPa (in Fig. 2) to 56 ± 8 MPa (in Fig. 4), but chopped alumina fiber did not change the brittle characteristics of the geopolymer samples. Correspondently the composite samples made by this chopped fiber reinforced resin showed higher "yielding strength" (defined as the point of deviation from the initially linear part of the flexure stress–strain curve) compared to those without chopped fibers, 61 ± 4 MPa

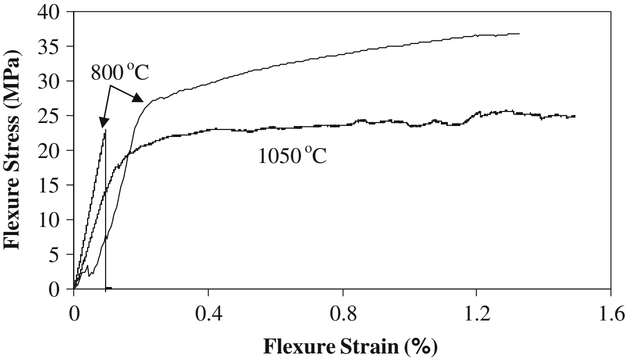


Fig. 5 Flexural stress–strain curves of geopolymer resin and geopolymer–stainless steel mesh composites after high temperature exposure

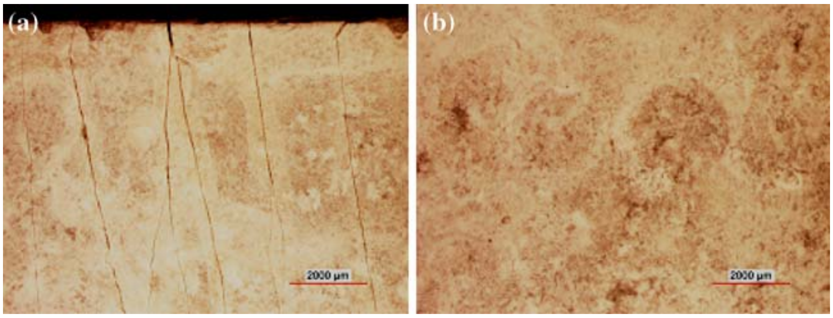
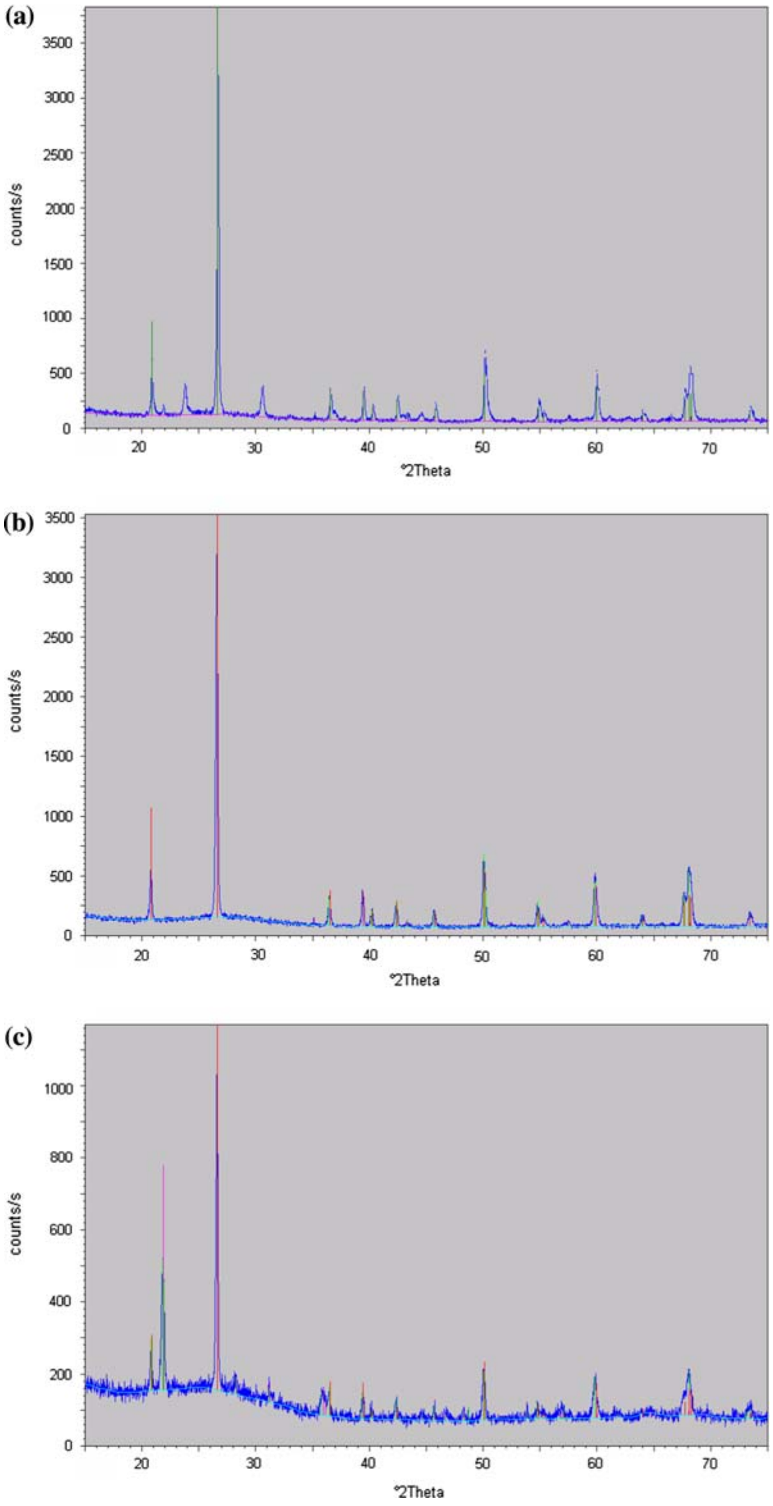


Fig. 3 Optical images of tensile (**a**) and compression (**b**) sides of a geopolymer–stainless steel mesh composite after a flexure test

Fig. 6 XRD plots of geopolymer resin after 80°C curing (**a**), 800°C/30 min (**b**), and 1,050°C/2 h (**c**) exposure



(in Fig. 4) versus 41 ± 5 MPa (in Fig. 2), and the maximum stress was almost 90 MPa, which indicates that in the composite structure the matrix or the interaction between the matrix and reinforcement contributes significantly to the strength, and the ductility is contributed by the steel mesh.

Flexure behavior of high temperature exposed composites

Figure 5 shows the room temperature flexure behaviors of the geopolymer–steel mesh composites after 800°C/30 min and 1,050°C/2 h treatment. The

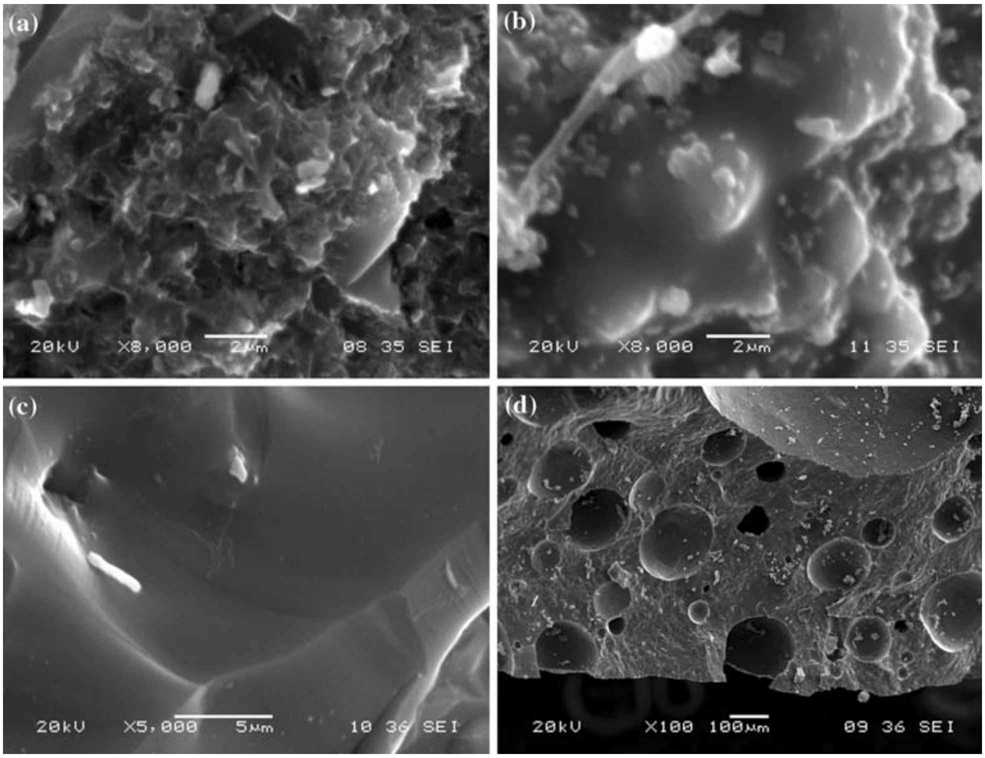


Fig. 7 SEM images of geopolymer resin after 80°C curing (**a**), 800°C/30 min (**b**), and 1,050°C/2 h (**c**, **d**) exposure

"yielding strength" of the high temperature exposed composite samples decreased compared to that of as-processed samples (average of 41 ± 5 MPa at 80°C, 24 ± 2 MPa at 800°C, and 18 ± 4 MPa at 1,050°C). But the composite samples can maintain their ductile behavior regardless of the treatment temperature, which is shown by the large flexure strain in Fig. 5. Interestingly the flexure strength of the geopolymer resin itself did not show significant degradation after 800°C/30 min exposure (see Figs. 2, 5). The flexure strength data for the geopolymer resin after 1,050°C/2 h treatment was not available because the samples were slightly melted and stuck on the bottom of the crucible, which indicated that this temperature was probably over the safe usage limit of the geopolymer matrix resin.

Figure 6 shows the XRD plots of the geopolymer resin after 80°C curing, 800°C/30 min exposure, and 1,050°C/2 h exposure. Since quartz (major) and corundum (minor) were used as the raw materials to react with potassium silicates for the geopolymerization reaction in this study, the XRD result of 80°C cured sample (Fig. 6a) shows strong quartz peaks, weak corundum peaks, and another two small peaks around 24 and 31°, which cannot be identified by the XRD software. These two unknown peaks should belong to potassium related phases. The XRD result indicates that not all silica and alumina were reacted with potassium silicates to form amorphous or semi-crystalline geopolymers. After 800°C/30 min exposure (Fig. 6b) the XRD plot only contains quartz and corundum peaks and the two unknown peaks disappeared, which showed that geopolymerization was enhanced and all the potassium was reacted to form amorphous materials. After 1,050°C/2 h exposure (Fig. 6c) corundum phase disappeared and the quartz started to transform to cristobalite. No potassium related crystalline phases were visible after 1,050°C, which was different from the results reported by Barbosa and MacKenzie [9] and they claimed kalsilite ($KAlSiO_4$) and leucite ($KAlSi_2O_6$) phases after 1,000 and 1,200°C treatment, respectively. Figure 7 shows the microstructure of geopolymer resin exposed to different temperatures. After 80°C curing, the resin had a composite structure, fine particles embedded in the bonding phase (Fig. 7a). Considering the XRD result in Fig. 6a, it seems reasonable to say that the particles are un-reacted quartz and corundum and the bonding phase is the geopolymer. After higher temperature exposure (Fig. 7b, c) apparently the bonding phase was more and became glassy and denser. On the other hand, compared to samples cured at 80°C, the high temperature exposed geopolymer resin showed more and larger voids (Fig. 7d). These microstructure evolutions coincide well with the observation of Lyon et al. [5]: the voids were created by the dehydration

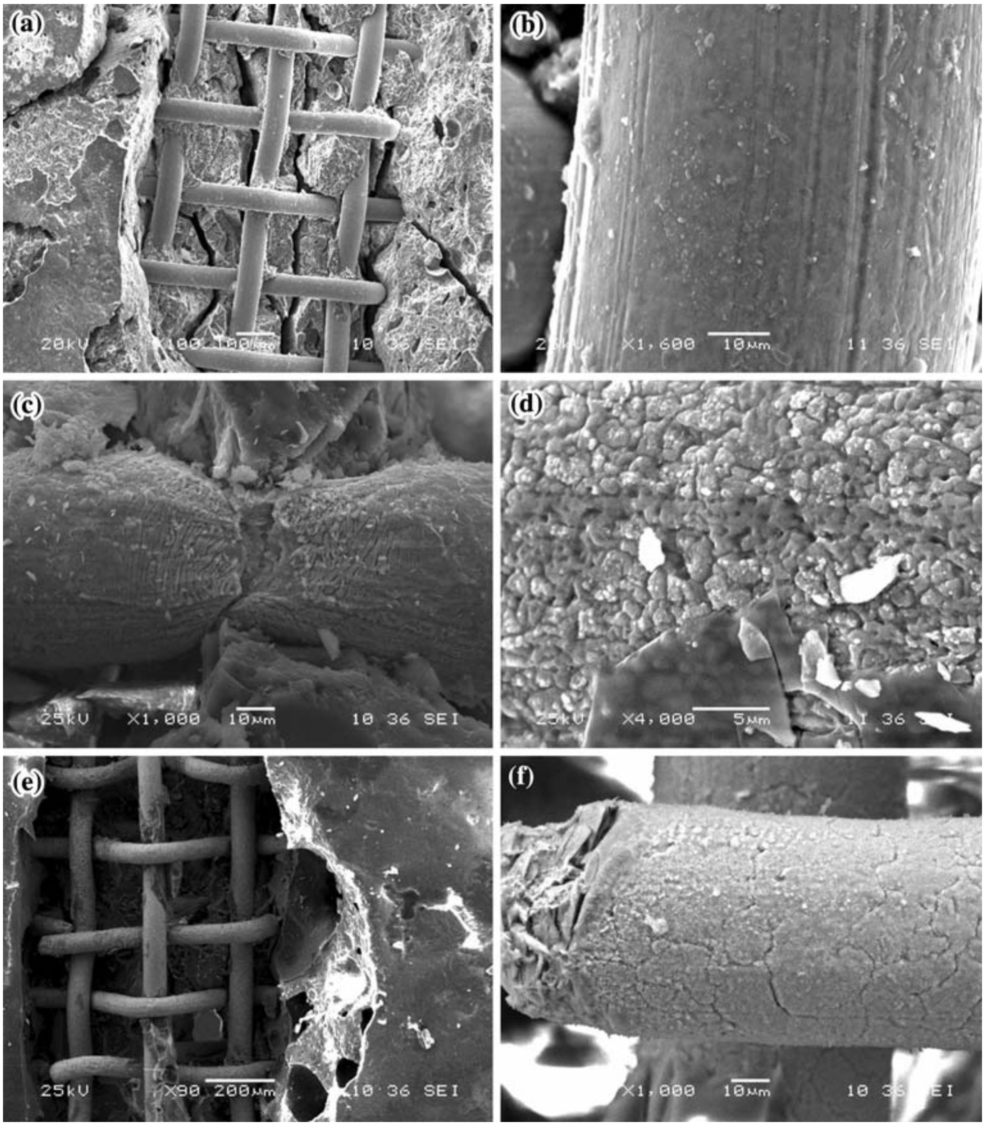


Fig. 8 SEM images of geopolymer–stainless steel mesh composites after 80°C curing (**a**, **b**), 800°C/30 min exposure (**c**, **d**), 1,050°C/2 h (**e**, **f**) exposure and then tested under flexure conditions (*left column* shows the composite structure and *right column* shows the surface of steel mesh)

process, i.e., water was liberated due to further cross linking of the geopolymer structure and water initially associated with the charge-balancing cation, potassium, escaped from the structure at high temperatures, meanwhile the geopolymer became stronger due to the further cross-linking. This twofold effect of high temperature exposure may explain why the 800°C exposed geopolymer resin showed similar flexure strength as the 80°C cured one.

However, for the composite samples, the high temperature exposure made stainless steel meshes to degrade (refer to Fig. 8). In Fig. 8 it is clearly shown that the surface of stainless steel mesh started to form rough scales and defects (Fig. 8d, f) after high temperature exposure and some meshes were broken during the flexure test (Fig. 8c, e), which was not seen in the 80°C cured sample (Fig. 8a, b). So it is not surprising for the high temperature exposed composites to show lower strength than the 80°C cured one. However, considering there is still sufficient strength and good ductility left even after 1,050°C/2 h exposure, the geopolymer/steel mesh composite samples are strong candidates for certain applications where catastrophic failure upon fire/high temperature exposure is a big concern.

Strength degradation at 800°C was also observed for the geopolymer resin and composites made with chopped alumina fiber reinforcement, but again the composite with stainless steel mesh maintained the good ductility even after the 800°C/30 min exposure (see Fig. 9). It is expected that by carefully selecting fire/high-temperature resistant geopolymer matrix and

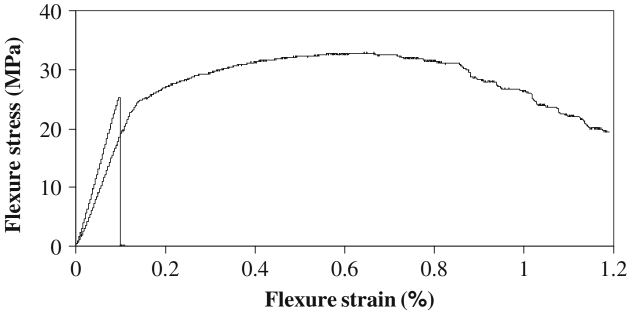


Fig. 9 Flexure stress and strain curves of chopped alumina fiber reinforced geopolymer resin and geopolymer–stainless steel mesh composite after 800°C/30 min exposure

metal meshes, composite materials with superior high temperature strength/toughness can be fabricated using the processing route reported here.

Conclusion

Novel low cost composites that showed graceful failure in flexure were fabricated by infiltrating stainless steel mesh with geopolymer resin. Four point flexure tests on these composites showed metal-like yielding behavior instead of catastrophic failure.

1. The ''yield strength'' was significantly higher than the flexure strength of the geopolymer matrix, after which the samples behaved like ductile materials.
2. While the brittle matrix material failed at ~0.1% flexure strain, complete fracture of composites did not occur even at >1% flexure strain.
3. Exposure to high temperatures of 800 and 1,050°C, resulted in the ''yield strength'' decreasing to ~59 and ~44% level respectively relative to the strength of as-processed samples. However, good ductility was still retained regardless of the high temperature treatment.
4. Using alumina chopped fiber to strengthen the geopolymer matrix material can further increase the ''yield strength'' of the composites.

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