The Effect of Si/Al on Mechanical Properties and Fracture Behavior of Stainless Steel Mesh/Cr$_p$ Reinforced Geopolymer Composites

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Abstract. In this study, a series stainless steel mesh/Cr$_p$ reinforced geopolymer composites with different Si/Al molar ratio (**N**) were designed and prepared, where **N** = 1.75, 2 and 2.25, respectively. The effect of Si/Al molar ratio in the geopolymer matrix on mechanical properties and fracture behavior of the geopolymer composites were investigated. The microstructure of geopolymer became more compact when Si/Al increased from 1.75 to 2, which was beneficial to the improvement of geopolymer's mechanical properties. And continuing to rise to 2.25 for Si/Al, the completely curing of geopolymer composites required more time compared with lower Si/Al, which can be attributed to the different microstructure and chemical composition caused by the different Si/Al. The optimum Si/Al molar ratio was about 2 at which the composites samples present the best mechanical properties with the flexure strength of 115.3 MPa and elastic modulus of 11.0 GPa, respectively. The results of fracture behavior suggested that geopolymer composites with *N* is 2.25 displayed the behavior characteristics of metal materials, which can be attributed to a poor integrated condition in interface between reinforcements and geopolymer matrix.

1 Introduction

While traditional design and evaluation approaches are based on the principle of maximizing the economic efficiency and include quality, cost, and time, the new approach of 'sustainable construction' emphasizes the importance of reducing the environmental impact of buildings and infra-structures [1]. Therefore, the reduction of CO_2 emissions and energy consumption in the cement industry has recently become a contentious issue, which corresponds to approximately 7% of the total emissions into the earth's atmosphere. In an effort to limit the environmental impact of concrete materials, there is increasing interest in the development an

environmental-friendly material. As an outstanding candidate, geopolymer can provide comparable performance to traditional cementitious binders in a range of applications, but with the added advantage of significantly reduced greenhouse emissions [2-7].

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Geopolymers are a class of cementitious aluminosilicate binder materials synthesized by a geopolymerization reaction between alumino-silicate oxides and alkali-metal silicate solutions under alkaline conditions, with partially or fully amorphous polymeric structures containing tetrahedral SiO_4 and AlO_4 randomly distributed along the polymeric chains [8-11]. The researches on the geopolymerization mechanism in this system are attracting more and more attentions, and the process may be summarized briefly as follows: dissolution, rearrangement, condensation and re-solidification [12-18]. The structure and mechanical properties of geopolymers are affected by several parameters such as chemical composition and reactivity of the raw materials, nature and concentration of the activating solution, curing conditions. The Si/Al molar ratio has a significant effect on the structural and mechanical performance. An optimum Si/Al ratio increases the strength of the resulting geopolymer. Apart from the ideal ratio, some losses may occur in the mechanical properties of the material due to the change of the geopolymer structure [19].

In this paper, a series stainless steel mesh/Cr_p reinforced geopolymer composites with different Si/Al molar ratio (***N***) were designed and prepared, where ***N*** = 1.75, 2 and 2.25, respectively. The effect of Si/Al molar ratio in the geopolymer matrix on mechanical properties and fracture behavior of the geopolymer composites were investigated.

2 Experiments

2.1 Material and synthesis

Geopolymers resin with composition of Si/Al = ***N***, 1.75, 2 and 2.25, respectively, were obtained by mixing metakaolin powder with potassium silicate solution. The metakaolin was prepared by calcining kaolin at 800 ºC for 2 h and the potassium silicate solution was prepared by dissolving a certain amount of amorphous silica (Shanghai Dixiang Indus, China) into KOH (Tianjin Fuchen Indus., China) solution stirring for 48 h in order to dissolve the silica completely. Then the metakaolin powder and chromium powder (40% mass ratio of metakaolin) were gradually added into the potassium silicate solution under ultrasonic-assisted high-shear mixer. To prepare composite samples, seven stainless steel mesh layers (Ø 70 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 3 d at 60ºC. After demoulding the hardened geopolymer composites were further cured at the same temperature for another 2 d. The major ingredient of the stainless steel mesh is $Cr_{0.19}Fe_{0.7}Ni_{0.11}$. The Si/Al molar ratio was adjusted by adding extra amorphous silica, and the amount of amorphous silica was calculated according to the Si/Al ratio.

2.2 Characterization

The phases of geopolymer composites were characterized using an X-ray diffractometer (XRD: Rigaku, D/MAX-2200VPC, Tokyo, Japan) with CuKa radiation at a scan rate of 4 º/min. Flexural strength measurements were conducted on specimens (4 mm×3 mm×36 mm) using a three-point-bending fixture on an Instron-500 testing machine with a span length of 30 mm at a crosshead speed of 0.5 mm/min. Tensile strength measurements were carried out on a Work of fracture was calculated by the area between load curves and X axis in the load/displacement curves till the load dropped to 90% of the maximum load. Six specimens were tested under each test condition. Fracture morphology of the geopolymer composites were investigated by a scanning electron microscope (SEM, 30 KV, Quanta 200 FEG, FEI Co., USA).

3 Results and discussion

3.1 Phase composition

Fig. 1 provided XRD patterns of geopolymer composites with different Si/Al ratio. As can be seen, all the samples show typical amorphous character, i.e., a broad amorphous hump at ~28º 2θ [20], which suggested that the different Si/Al ratio did not changed the nature of the geopolymer matrix. With the rise of Si/Al, the amount of SiO_2 in the composites decreased gradually. The results of XRD also indicated that the stainless steel mesh and chromium powder were not involved in the reaction with geopolymer matrix [21].
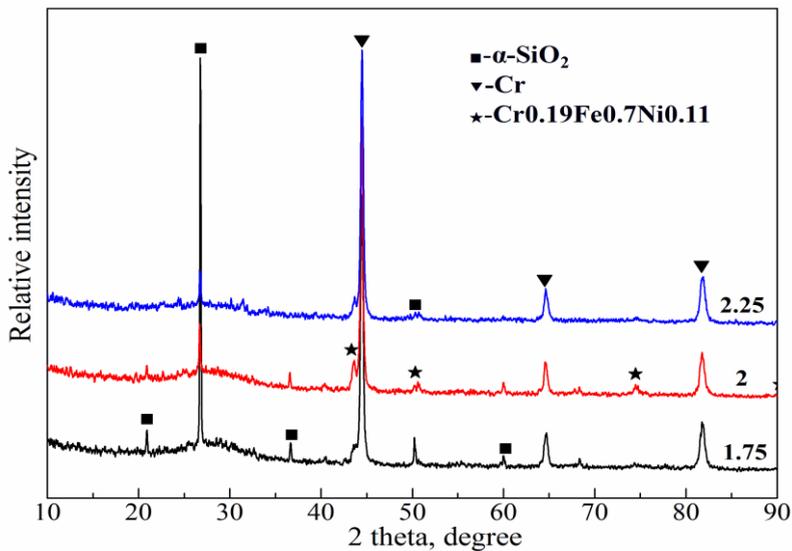

Fig. 1. XRD patterns of geopolymer composites with different Si/Al ratio.

3.2 Microstructure

Fig. 2 displayed the surface morphologies of geopolymer composites with different Si/Al ratio. The surface morphology of geopolymer composites became much more compact and smoothly when *N* increased from 1.75 to 2. When *N* =1.75, the viscosity of geopolymer slurry was relatively low, which favored the escape of the water in the initial stage of curing process, which can be explained the pore in the samples surface (Fig. 2c). Meanwhile, more surface water and pore water will loss in the curing process due to the loose microstructure. However, a geopolymer slurry with a moderate viscosity of was obtained when the *N* = 2 compared with that of 1.75 [22, 23]. Therefore, more uniform and compact microstructure was observed in the samples surface (Fig. 2b). When *N* came to 2.25, there were significant difference with *N* = 1.75 or 2. After 3 d curing, the geopolymer gel has not completely solidified, which was attributed to the presence of massive potassium silicate [24]. There were many cracks in the samples surface with *N* = 2.25, which may be caused by the extrusion in the demoulding process.
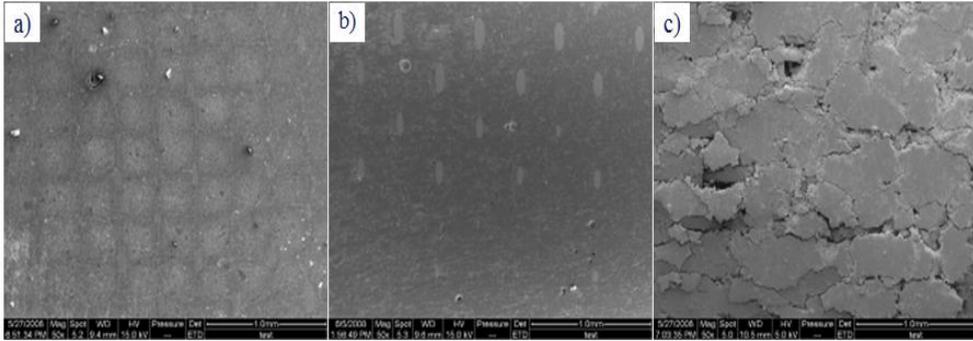
Fig. 2. Surface morphologies of geopolymer composites with different Si/Al ratio, a) 1.75, b) 2 and c) 2.25

3.3 Mechanical properties

The Si/Al ratio has a great influence on the mechanical properties of the resulting geopolymer composites, as shown in Fig. 3. With the increase in Si/Al ratio, flexural strength, elasticity modulus, compressive strength and work of fracture of the composites showed similar trends, i.e., increasing first, reaching the highest value at $N = 2$, and then decreasing. Take flexural strength for example (Fig .4): it increased from 96.3 MPa for composites with $N = 1.75$ to the highest of 15.8 MPa for composites with $N = 2$. And almost all the mechanical properties reached the peak value when $N = 2$. However, there was a significant declined in performance when $N = 2.25$. As shown in Fig. 4 d, a mass of geopolymer matrix flaked away. The surface of stainless steel mesh was still smooth and no corrosion in the alkaline environment [21]. This might result from the weak interfacial strength between stainless steel mesh and geopolymer matrix [25]. In addition, microscopic defects such as interfacial gaps or the cracks in the surface also caused the loss of mechanical properties of geopolymer composites [26].
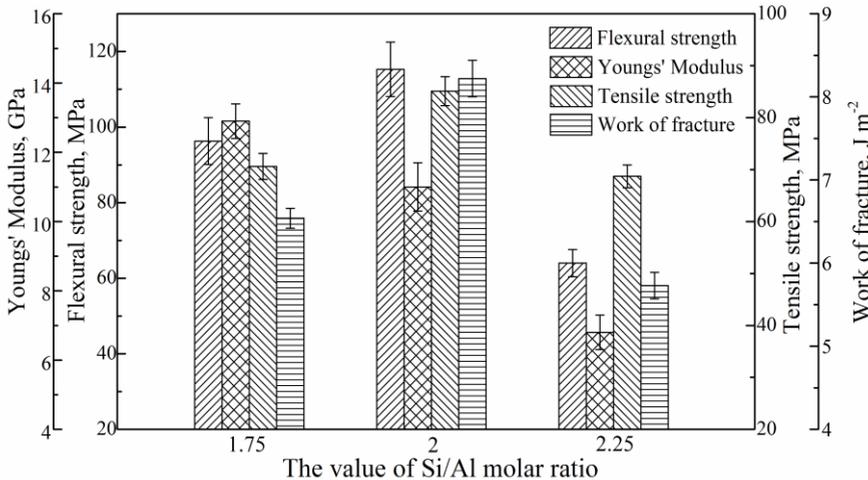

Fig. 3. Mechanical properties of geopolymer composites with different Si/Al ratio, a) 1.75, b) 2 and c) 2.25.
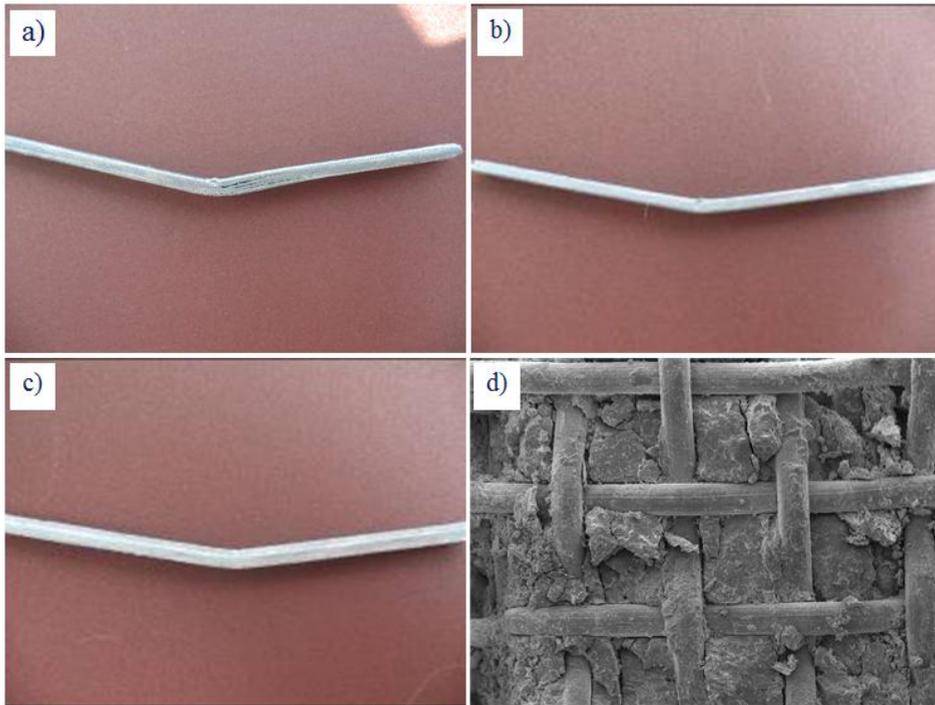
Fig. 4. The samples after three-point bending tests, a) 1.75, b) 2, c) 2.25 and
d) the surface morphology with $N = 2.25$.

3.4 Fracture behavior

Fig. 5. shows the load-displacement curves of geopolymer composites with different Si/Al
molar ratio. Unlike $N = 2.25$, the samples with $N = 1.75$ or 2 showed a non-linear elastic
increase of load before reaching the maximum load values. Then, the load decreased slowly
with the displacement. The reason may be attributed to the bridging and sliding after
debonding and pulling-out of the stainless steel mesh from the weak geopolymer matrix [27].
Meanwhile, the presence of chromium powder also was beneficial to enhancement of
composites' toughness. However, the samples with $N = 2.25$ displayed a fracture behavior as
similar with metal materials, which indicated that there was little interfacial strength between
stainless steel mesh and geopolymer matrix. The change of work of fracture also suggested
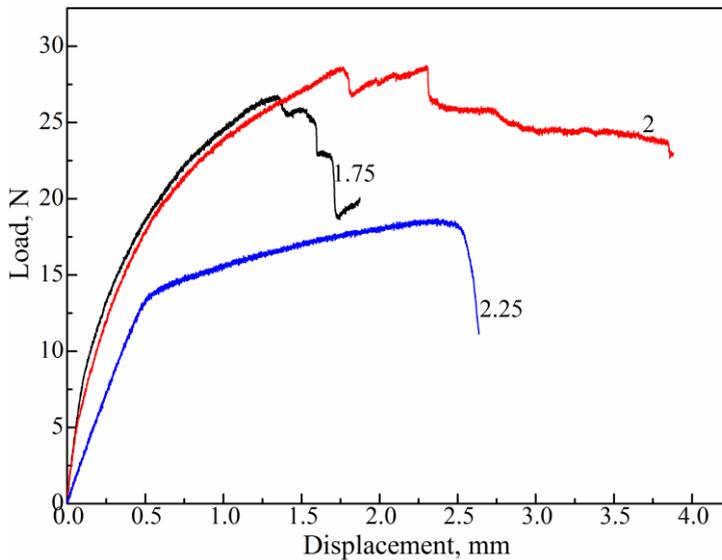the optimum Si/Al ratio should be 2.

Fig. 5. Fracture behavior of geopolymer composites with different Si/Al ratio.

4 Summary

In this paper, the effect of Si/Al molar ratio on the performance of stainless steel mesh/Cr$_p$ reinforced geopolymer composites was investigated. The results indicated that Si/Al molar ratio had a significant impact on the final microstructure and mechanical properties of resulting geopolymer composites. The microstructure of geopolymer became more compact when Si/Al increased from 1.75 to 2, and the corresponding mechanical properties also reached the peak value. However, when Si/Al came to 2.25, the mechanical properties of composites declined. The results of fracture behavior suggested that the samples with $N =$ 2.25 displayed a fracture behavior as similar with metal materials, which different with $N =$ 1.75 and 2. The reason can be ascribed to different interfacial strength between reinforcements and geopolymer matrix, and composites with $N = 2$ has an optimum interfacial strength compared with $N =$ 1.75 and 2.25.

References

1. K.H. Yang, J.K. Song, K.I. Song, J. Clean. Prod., **39**, 265 (2013)
2. P. Hlavacek, V. Smilauer, F. Skvara, L. Kopecky, R. Sulc, J. Eur. Ceram. Soc., **35,** 703 (2015)
3. A. Favier, G. Habert, J.B.D.E.d. Lacaillerie, N. Roussel, Cem. Concr. Res., **48**, 9 (2013)
4. Z. Zhang, X. Yao, H. Zhu, Appl. Clay Sci., **49**, 1 (2010)
5. V.F.F. Barbosa, K.J.D. Mackenzie, Mater. Res. Bull., **38**, 319 (2003)
6. F. Slaty, H. Khoury, H. Rahier, J. Wastiels, Appl. Clay Sci., **104**, 229 (2015)
7. W.D.A. Rickard, L. Vickers, A.V. Riessen, Appl. Clay Sci., **73,** 71 (2013)
8. J. Davidovits, J. Therm. Anal., **37**, 1633 (1991)
9. J. Davidovits, J. Therm. Anal., **35,** 429 (1989)

10. K. Komnitsas, D. Zaharaki, A. Vlachou, G. Bartzas, M. Galetakis, Adv. Powder Technol, **26**, 368 (2015)
11. B.H. Mo, Z. He, X.M. Cui, Y. He, S.Y. Gong, Appl. Clay Sci., **99**, 144 (2014)
12. J.L. Provis, J.S.J.V. Deventer, *Geopolymers : Structures, Processing, Properties and Industrial Applications* (Elsevier, United Kingdom, 2009)
13. X. Yao, Z. Zhang, H. Zhu, Y. Chen, Thermochim Acta, **493**, 49 (2009)
14. L. Weng, K.S. Crentsil, J. Mater. Sci., **42,** 2997 (2007)
15. P.S. Singh, M. Trigg, I. Burgar, T. Bastow, Mater. Sci. Eng. A Struct., **396**, 392 (2005)
16. M.R. Rowles, B.H. O'Connor, J. Am. Ceram. Soc., **92**, 2354 (2009)
17. I. Ozer, S.S. Uzun, Ceram. Inter., **41**, 10192 (2015)
18. K. Komnitsas, D. Zaharaki, V. Perdikatsis, J. Mater. Sci., **42**, 3073 (2006)
19. G. Gorhan, G. Kurklu, Compos. Part B-Eng., **58**, 371 (2014)
20. P. Duxson, A.F. Jimenez, J.L. Provis, G.C. Lukey, A. Palomo, J.S.J.V. Deventer, J. Mater. Sci., **42**, 2917 (2006)
21. P.G. Allison, C.A. Weiss, J.R.D. Moser, A.J. Diaz, O.G. Rivera, S.S. Holton, Compos. Part B-Eng., **78**, 131 (2015)
22. P. Duxson, G.C. Lukey, J.S.J.V. Deventer, J. Non Cryst Solids, **352,** 5541 (2006)
23. P. Duxson, G.C. Lukey, J.S.J.V. Deventer, Langmuir, **22**, 8750 (2006)
24. A. Hajimohammadi, J.L. Provis, J.S.J.V. Deventer, Cem. Concr. Res., **41**, 210 (2011)
25. A. Castel, S.J. Foster, Cem. Concr. Res., **72**, 48 (2015)
26. J. Yuan, P. He, D. Jia, S. Yan, D. Cai, L. Xu, Z. Yang, X. Duan, S. Wang, Y. Zhou, Ceram. Inter., **42**, 5345 (2016)
27. Q. Zhao, B. Nair, T. Rahimian, P. Balaguru, J. Mater. Sci., **42**, 3131 (2007)