Influence of Plasma Treatment on the Flexural Properties of Geopolymer Composites

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Abstract

In recently years, geopolymer or polysialate composites have been developed and applied for a variety of fields. They are known as inorganic matrices composites, which are fabricated and cured at room temperature or thermoseted in a simple autoclave. Geopolymer composites provide an excellent opportunity for achieving the goal of producing low cost high temperature resistant composite. The aim of this study is an investigation and comparing the flexural properties of geopolymer composites after reinforcements were influenced by plasma treatment with original fibre fabric. And preliminary results of the improvement in tensile strength, Young's modulus, elongation of E-glass fibre fabric due to plasma treatment are studied in this paper.

Many reinforcements and geopolymer matrices are now available, but our works are focused on geopolymer composite Q17, Q13K-1 combination with E-glass fibre fabric which was investigated, fabrication procedures together with their specific properties of these materials have been evaluated, compared and discussed.

Keywords: E-glass fibre fabric, plasma treatment, flexural strength, geopolymer, surface treatment.

1. Introduction

In recent years, geopolymer materials have attracted much more attention due to their excellent fire resistance, low density, low cost, low curing/hardening temperatures, easy processing, excellent mechanical properties, environmentally friendly nature, long-term durability, heavy metal ions fixation and acid resistance [1-3]. The properties of metakaolin-based geopolymer are directly impacted not only by the specific surface and composition of initial metakaolin and the type, composition and relative amount of alkali activator used but they also depend on the conditions during the initial period of geopolymerization reaction [4].

E-glass fibre fabric 486g/m^2 RT 490 is a bidirectional fabric made by interweaving direct roving in plain weave pattern. E-glass fibre fabric is compatible with many resins. It is a high-performance reinforcement and widely used in hand and machine production, such as boats, vessels, plane and automotive parts, densely aligned fibers resulting in a high strength. Good mold ability and drape ability making handing easy. Warp and weft roving aligned in a parallel, flat manner resulting in uniform tension, very little twist, excellent roll out characteristics, good wet-out in resins [5].

In this paper, we developed a kind of E-glass fibre fabric performs with the help of the plasma treatment for reinforcing geopolymer matrix. In addition, effects of methane plasma treatment and number of layers (from 5 to 7 layers) on the mechanical properties, Young's modulus and elongation of composites were systematically investigated.

2. Experimental

2.1 Properties of E-glass fibre fabric

Fibre E-glass in fabric form offers an excellent combination of properties from high strength to fire resistance. Wide ranges of yarn sizes and weave patterns provide huge number of design potential allowing the end user to choose the best combination for material performance, economics and product flexibility. E-glass fibre is an inorganic material and will not burn or support combustion. It retains approximately 25% of its initial strength at 540^{o}C. E-glass fibre does not stretch or shrink after exposure to extremely high or low temperatures. Additions, It does not absorb moisture, and does not change physically or chemically when exposed to water. However, the increased surface area makes them much more susceptible to chemical attack because of their high ratio of surface area to weight. By trapping air within them, blocks of E-glass fibre make good thermal insulation, with a thermal conductivity on the order of 0.05 W/(mK). E-glass fibre is an excellent material for electrical insulation and low coefficient of thermal expansion. E-glass strength is usually tested and reported for "virgin" fibre: those which have just been manufactured. The freshest, thinnest fibre is the strongest because the thinner fiber is more ductile. The more the surface is scratched, the less the resulting tenacity. Because E-glass has an amorphous structure, its properties are the same along the fibre and across the fibre. Humidity is an important factor in the tensile strength. Moisture is easily adsorbed, and can worsen microscopic cracks and surface defects, and lessen tenacity [6, 7].

Fig. 1 E-glass fabric 486 g/m^2 RT 490

2.2 Optimization parameters of plasma

Radio frequency (RF) plasma reactor was employed for the plasma surface treatment of fibre. The RF energy is supplied to the system through an isolated electrode, which is powered by a 13.56 MHz (1200 W maximum power) home-made matching network. The reaction chamber is made from stainless steel tubing, 1200 mm in height and 280 mm inner diameter. The system is controller for gas inlet, a pressure gauge, and a rotary vacuum pump.

The process of plasma treatment is described as follows: These fibres were placed on the substrate holder inside the deposition chamber and were exposed to methane low-pressure glow discharge plasma in the 5 ÷ 30 min range. A methane flow rate from 10 sccm to 30 sccm was used, and the working pressure inside the plasma chamber was maintained in the 25 ÷ 40 Pa range. Negative bias voltage (V_b) was varied from 300 to 900 V [8].

2.3 Specimen preparation

The samples were prepared using a standard vacuum bagging technique. The fabrics were impregnated with the matrix by-hand, stacked together and placed in the vacuum bag. The samples were cured using a technique called "press vacuum bagging" (-1atm and room temperature 20^{o}C) for 2 hour. The bag was placed in a heated press at -1atm in the furnace at 80^{o}C for 2 hours. The bag was then removed from the press and inserted into a furnace for final curing at 80^{o}C for 20 hours or until a constant mass was achieved. The plates were approximately (110 x 100) mm in plan dimension. The coupons were cut from the sample using a high speed diamond blade. They were then inspected to ensure that the thickness was constant throughout the plate. The volume fraction of reinforcement was approximately 50%.

2.4 Flexural Testing

The sample dimensions were tested under three-point bending in accordance with ISO 178. The flexural tests were conducted over a simply supported span length of 64 mm with a center-point load by the testing machine INSTRON Model 4202 (maximum load of the sensor: 10.000 N) and a crosshead speed of 2 mm/min.

The Young's modulus and flexural strength were calculated from this test. Flexural strength (R_{mo}) was calculated using:

$$R_{mo} = \frac{3FL}{2bh^2} \quad (1)$$

Where L is the span between the two supports; F is the maximum load; b is the width of the sample; h is the thickness of the sample.

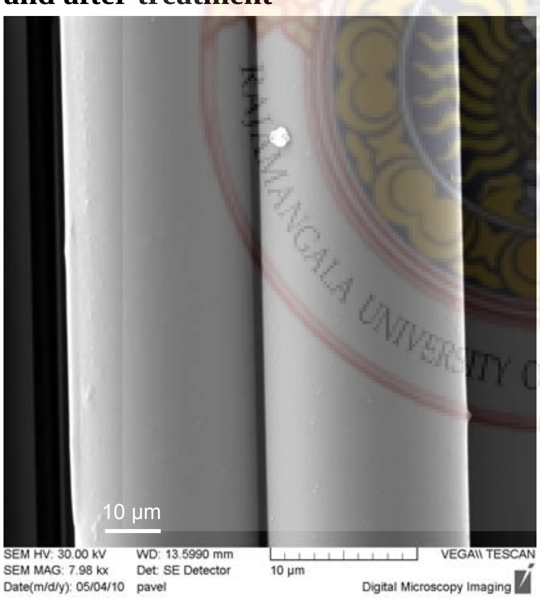
Surfaces of the fibre fabric and the composites were observed by scanning electron microscopy.

3. Results and discussion

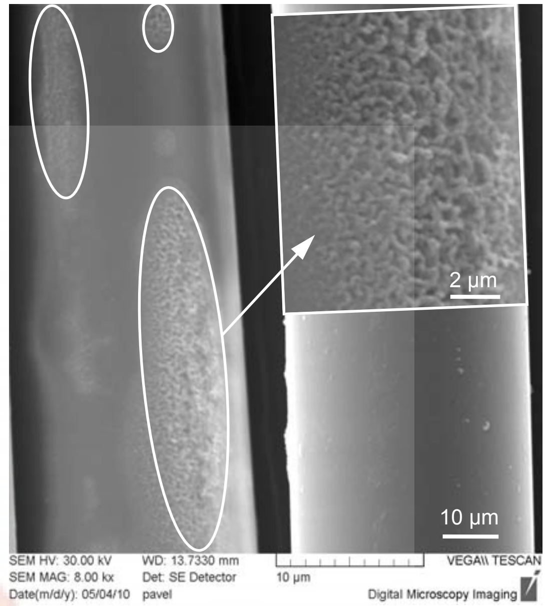
3.1 Optimization parameters of plasma

There were four main plasma-treatment parameter effects to mechanical properties on the surface chemical composition of basalt and E-glass fibre fabric: treatment time, flow rate of methane, pressure and bias voltage [8]. After plasma treatment, single filament of each fibre were separated with a magnifier and prepared on a punched mounting tab. The single filament test piece was bonded by adhesive so as to let the length specified gauge length under the condition to make the filament straight along the center line of the mounting tab. This was evaluated in accordance with Japanese Industrial Standard (JIS R 7601). Tensile strength and Young's modulus were calculated from the load-elongation records and the cross-sectional area measurements [9]. The authors determined the treatment time of the fibres were optimized at around 5 min, while the flow rate of methane was CH_4 = 30 sccm, bias voltage V_b = 500 V and pressure p = 30 Pa.

3.2 The morphologies of the surfaces of E-glass fibre fabric before and after treatment



(a)



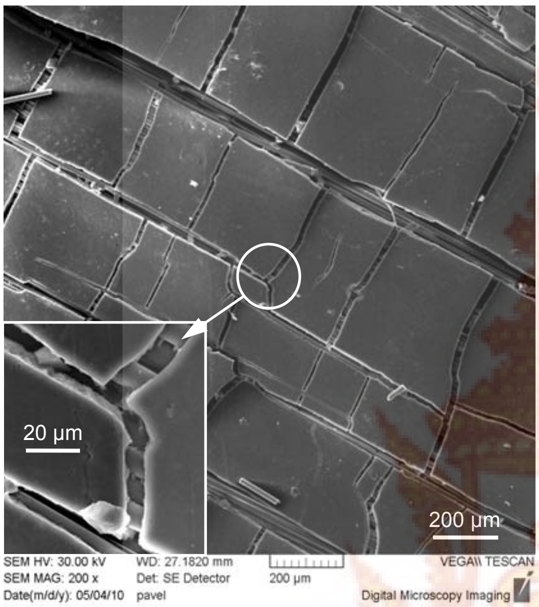
(b)

Fig. 2 SEM images surface of E-glass fibre fabric: *(a)* Untreated, *(b)* Plasma treated

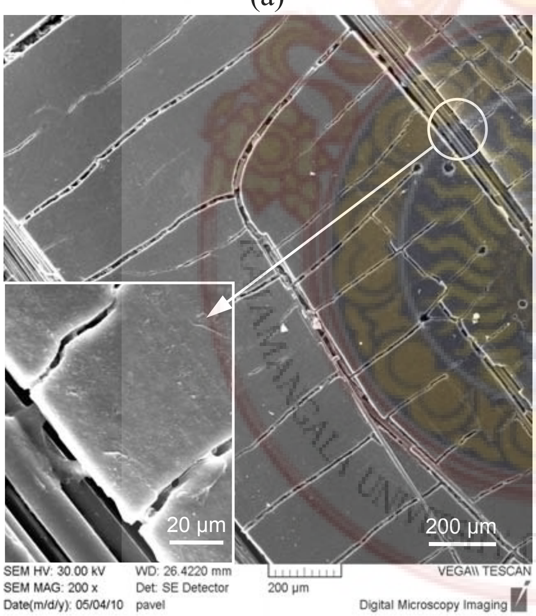
The SEM photographs of E-glass fibre fabric are collected in Figure 2. Fig. 2a shows the surfaces of the untreated E-glass fibre have smooth surfaces with clear longitudinal grains, and the plasma treated fibre in Fig. 2b shows that these surfaces of E-glass fibre had a rougher surface than the original fibre. This is believed to be one of the main reasons to improve the fibre and matrix adhesion.

3.3 The morphologies of the surfaces of composite reinforced with E-glass fibre fabric before and after treatment

The propagation of cracks in reinforced materials is determined by the strength and deformation characteristics of the matrix and the fibre, the strength of their bond at the boundary, surface defects, the form of loading, and other factors. By choosing in the appropriate manner modifying of reinforced systems it is possible to reduce of cracks. Figure 3a shows that micro-cracks on the surface of composite larger than figure 3b. In this paper, we suppose that the crack length is almost equivalent to the representative dimension of the microscopic heterogeneity. The effect of the heterogeneity on the crack behaviors must be considered.



(a)



(b)

Fig. 3 SEM images surface of geopolymer composite reinforced with E-glass fibre fabric: *(a)* Untreated fibre*,* *(b)* Plasma treated fibre

3.4 Mechanical properties of geopolymer composite

The flexural strength, Young's modulus and elongation values of untreated and plasma-treated E-glass fibre fabric geopolymer composites were measured during the testing. Average results are presented in table 1 and table 2. Comparison between plates made on geopolymer composite Q17, Q13K-1 combination with untreated E-glass fibre fabric and plasma treated E-glass fibre fabric are shown in Figure 4 and Figure 5.

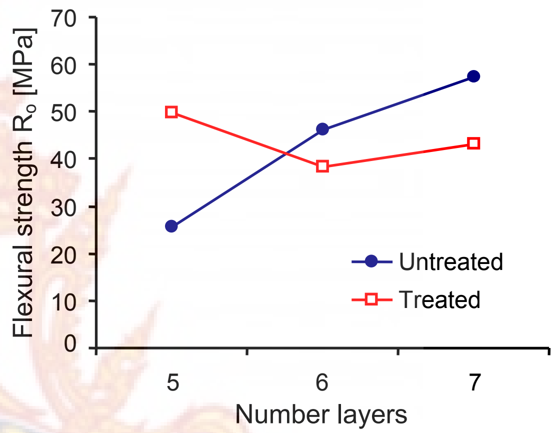


Fig. 4 Effects of plasma treatment fibre on flexural strength of geopolymer composite Q17 reinforced with E-glass fibre fabric

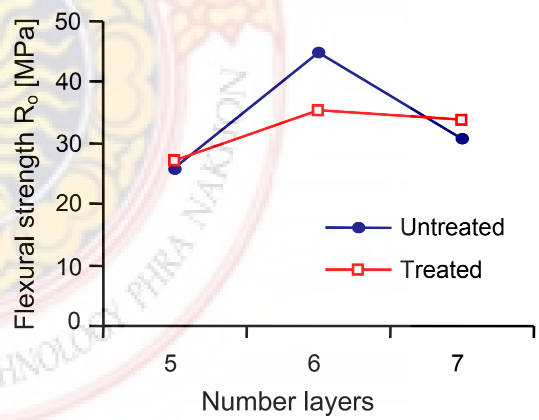


Fig. 5 Effects of plasma treatment fibres on flexural strength of geopolymer composite Q13K1 reinforced with E-glass fibre fabric

Table 1: Flexural properties of Q17 geopolymer matrix and E-glass fibre fabric

Number Layers	Untreated					Plasma treatment				
	Denisty (g/cm^3)	Thickness (mm)	A [%]	R_o [MPa]	E [GPa]	Denisty (g/cm^3)	Thickness (mm)	A [%]	R_o [MPa]	E [GPa]
5	1.77	1.85	1.78	25.52	6.63	1.73	1.72	1.39	49.87	15.98
6	2.06	2.38	1.87	46.28	15.27	2.04	2.36	2.18	38.18	11.32
7	1.83	2.82	1.60	57.33	9.53	1.78	2.71	1.40	43.08	9.29

Table 2: Flexural properties of Q13K1 geopolymer matrix and E-glass fibre fabric

Number Layers	Untreated					Plasma treatment				
	Denisty (g/cm^3)	Thickness (mm)	A [%]	R_o [MPa]	E [GPa]	Denisty (g/cm^3)	Thickness (mm)	A [%]	R_o [MPa]	E [GPa]
5	1.75	2.17	1.26	26.08	8.84	1.96	2.11	1.94	27.20	7.30
6	1.86	2.35	1.40	44.96	14.96	1.92	2.36	2.10	35.41	7.70
7	1.83	2.96	1.17	30.90	11.48	1.99	2.86	1.80	33.79	8.79

Figure 4 presents the Q17 geopolymer matrix reinforced with 5 layers untreated E-glass fibre fabric was determined the flexural strength to be 25.52 MPa. While the fibre was treated with plasma, the flexural strength value increased to 49.87 MPa. This indicates a 51% increase of flexural strength. Furthermore, the value of Young's modulus of plasma treated fibre composite was increased to greater than that of untreated fibre by approximately 41%. However, matrix reinforced with 6 layers and 7 layers fibre treated at plasma was decreased in both the flexural strength and Young's modulus.

In figure 5 shows that flexural strength values of the Q13K1 geopolymer matrix reinforced with 5 layers and 7 layers untreated fibre were determined in turn as 26.08 MPa, 30.90 MPa. While E-glass fibre fabric composite was treated at plasma, the increase slightly (about 10%) in the flexural strength for both case and reduced about 80% in Young's modulus comparing with untreated was observed. However, in 6 layers fibre composite were treated at plasma was decreased (about 78%) in the flexural strength.

4. Conclusions

The conclusions of the present research can be stated as follows:

The surfaces of the fibre are modified by methane plasma. Therefore, the interfacial bond state is modified, and the properties of the composite are improved. However, during the preparing and curing process, the authors have used the velocity to wetting fibre with the resin no rational for E-glass fibre fabric. In the future, we will find optimal curing conditions and the velocity to improve the adhesion between geopolymer matrix and reinforcements.

SEM images of the composite surface (Fig. 3), we can see that when we modified the fibre with plasma treatment, the micro-cracks seem smaller than original fibre. We can say that the adherence of silica-based geopolymer matrix with treated E-glass fibre fabric is better than untreated fibre, it will helps restrict micro-crack propagation because the micro-cracks in the matrix are determined as inborn defects of inorganic matrix composites.

In the future, the authors will apply this method to cure other fibre and changing methane plasma by nitrogen plasma treatment. Because composites prepared with surface plasma-treated fibre has shown generally much better wetting and adhesion than those made from untreated fibre and improved mechanical properties of the composites.

5. Acknowledgements:

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